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PB-272 722

# Studies of the Pressurized Fluidized-Bed Coal Combustion Process

Exxon Research and Engineering Co, Linden, N J

Prepared for

Industrial Environmental Research Lab, Research Triangle Park, N C

Sep 77

PB 272 722

EPA

U.S. Environmental Protection Agency
Office of Research and Development

Industrial Environmental Research
Laboratory
Research Triangle Park, North Carolina 27711

EPA-600/7-77-107
September 1977

# STUDIES OF THE PRESSURIZED FLUIDIZED-BED COAL COMBUSTION PROCESS

Interagency
Energy-Environment
Research and Development
Program Report

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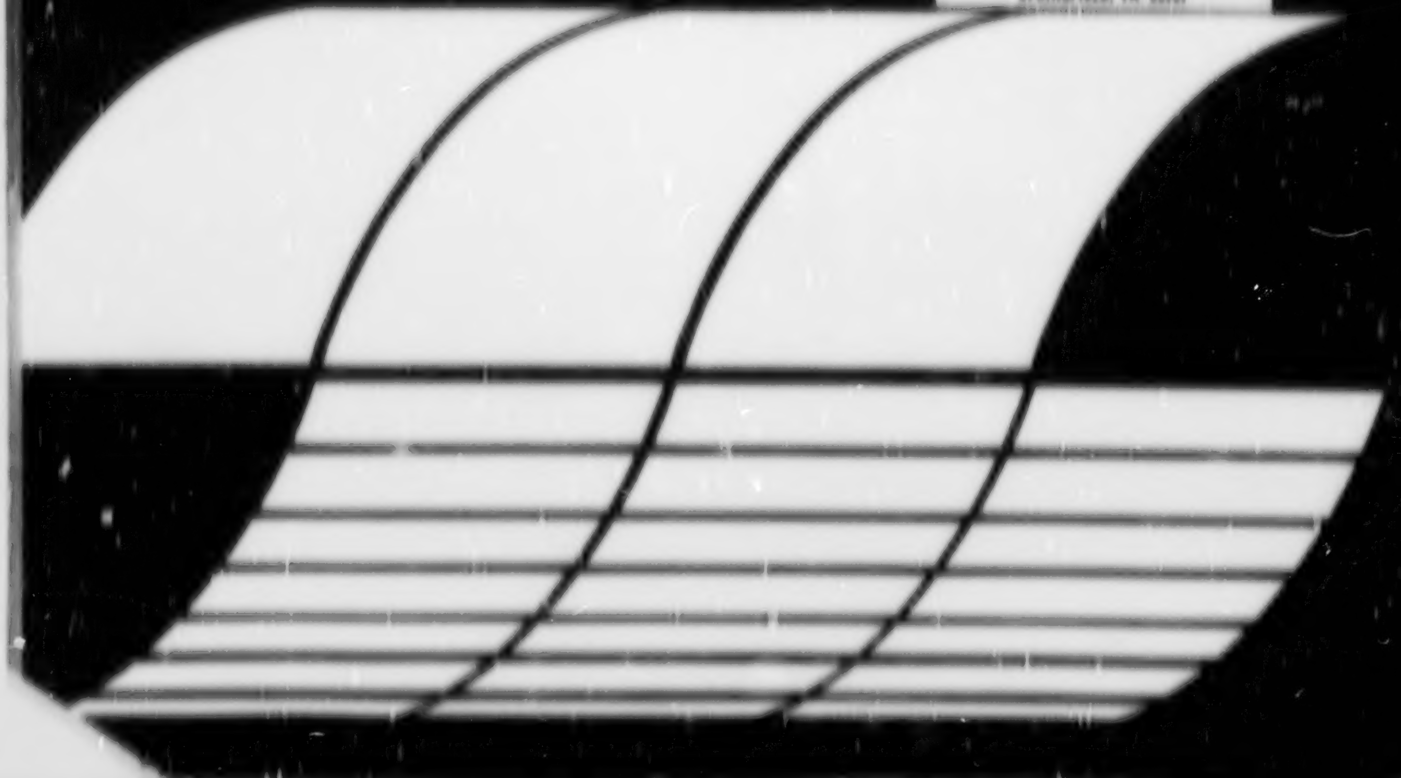

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

PB 272 722

1. REPORT NO.
EPA-600/7-77-107

2.

3. RECIPIENT'S ACCESSION NO.

4. TITLE AND SUBTITLE
Studies of the Pressurized Fluidized-Bed Coal Combustion Process

5. REPORT DATE
September 1977

6. PERFORMING ORGANIZATION CODE

7. AUTHOR(S)
R.C. Hoke, R.R. Bertrand, M.S. Nutkis, D.D. Kinzler, L.A. Ruth, M.W. Gregory, and E.M. Magee

8. PERFORMING ORGANIZATION REPORT NO.
EXXON/GRU.16GPGS.77

9. PERFORMING ORGANIZATION NAME AND ADDRESS
Exxon Research and Engineering Co.
P.O. Box 8
Linden, New Jersey 07036

10. PROGRAM ELEMENT NO.
EHE623A

11. CONTRACT/GRANT NO.
68-02-1312 and -1451

12. SPONSORING AGENCY NAME AND ADDRESS
EPA, Office of Research and Development
Industrial Environmental Research Laboratory
Research Triangle Park, NC 27711

13. TYPE OF REPORT AND PERIOD COVERED
Phase; 8/75-7/76

14. SPONSORING AGENCY CODE
EPA/600/13

15. SUPPLEMENTARY NOTES IERL-RTP project officer for this report is D. Bruce Henschel, Mail Drop 61, 919/541-2825. EPA-600/7-76-011 is the previous EPA report relating to this work.

16. ABSTRACT

The report gives results of studies of the environmental aspects of the pressurized fluidized-bed coal combustion process, using two experimental facilities: a 218 kg coal/hr "miniplant" continuous combustion/sorbent regeneration system (0.63 MW equivalent), and a 13 kg coal/hr "batch" combustion unit. Combustion studies were conducted to characterize the emissions of SO2, SO3, NOx, particulates, and CO from the combustors as a function of combustion conditions. Operating results from these combustion runs defined the dolomite and limestone sorbent feed rate required to keep SO2 emissions within the current EPA New Source Performance Standards for coal-fired utility boilers, considering the effects of coal sulfur content, sorbent type, gas residence time in the bed, bed temperature, bed pressure, and excess air. NOx emissions remained within the range 0.1-0.4 lb/million Btu, compared to the current standard of 0.70 lb/million Btu. Particulate emissions from the miniplant combustor, after two stages of cyclones, ranged from 0.8 to 4.2 lb/million Btu (mass median particle size of 7 microns), compared to the EPA standard of 0.10 lb/million Btu; thus an additional stage of particle control would be required. Emissions of SO3 and CO were generally low. Shakedown of the miniplant regenerator was completed, culminating with a 24-hr combustion/regeneration run during which sorbent was transferred continuously between the combustor and the regenerator.

17. KEY WORDS AND DOCUMENT ANALYSIS

| a. DESCRIPTORS | | b. IDENTIFIERS/OPEN ENDED TERMS | c. COSATI Field/Group |
|---|---|---|---|
| Air Pollution | Fluidized-Bed Processors | Air Pollution Control | 13B |
| Desulfurization | Combustion | Stationary Sources | 07A, 07D |
| Flue Gases | Regeneration (Engineering) | Fluidized-Bed Combustion | 21B |
| Limestone | | | 08G |
| Dolomite (Rock) | | Limestone-Based Desulfurization Process | |
| Calcium Oxides | | | 07B |

18. DISTRIBUTION STATEMENT
Unlimited

19. SECURITY CLASS (This Report)
Unclassified

20. SECURITY CLASS (This page)
Unclassified

21. NO. OF PAGES
214

22. PRICE
A10 - A01

EPA Form 2220-1 (9-73)

## RESEARCH REPORTING SERIES

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EPA-600/7-77-107
September 1977

# STUDIES OF THE PRESSURIZED FLUIDIZED-BED COAL COMBUSTION PROCESS

by

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Contract No. 68-02-1312 and -1451
Program Element No. EHE623A

EPA Project Officer: D. Bruce Henschel

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Research Triangle Park, N.C. 27711

Prepared for

U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
Washington, D.C. 20460

# ABSTRACT

The pressurized fluidized bed combustion of coal and regeneration of spent sorbent were studied in the continuous 480 lb coal/hr (220 kg/hr) "miniplant" unit. Supporting studies were also carried out in a smaller batch combustion unit. Emissions of $SO_2$, $SO_3$, $NO_x$, CO and particulates from the combustors were measured as a function of combustion conditions. Dolomite and limestone requirements needed to keep $SO_2$ emissions within new source performance standards were estimated based on the experimental results. $SO_3$ and CO emission levels were generally low. $NO_x$ emission levels were well within the current new source performance standard. Particulate emission exceeded the new source performance standard and will require the use of an additional particulate removal device beyond two stages of conventional cyclones to meet the emission standard. Carbon combustion efficiencies were also measured. Levels of 99% were achieved at higher combustion temperatures.

Shakedown of the miniplant regenerator section was completed by a run in which the combustor and regenerator were both operated and solids were transferred between the combustor and regeneration sections continuously for a 24 hr. period. A solids transfer system was developed prior to the run, permitting the continuous transfer of the hot solids between the combustor and regenerator.

Additional studies are planned to develop additional combustion data, to study the removal of particulates from the combustor flue gas at high temperature and high pressure, to demonstrate continuous combustion and sorbent regeneration for a 100 hr. period and make a comprehensive analysis of potentially harmful emissions from the miniplant unit.

This work was submitted in fulfillment of Contract Numbers 68-02-1312 and 68-02-1451 by Exxon Research and Engineering Company under sponsorship of the Environmental Protection Agency. Work was completed in July 1976.

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# ACKNOWLEDGEMENTS

The authors wish to express their appreciation to the many individuals who played major roles in the conduct of this program at Exxon Research and Engineering Company. In particular, we wish to acknowledge the efforts of H. R. Stiskowski, the miniplant operations supervisor. His contributions played a large part in the successful operation of the miniplant. We also wish to acknowledge the efforts of the operating crew, T. C. Gaydos, R. E. Long, G. E. Walsh, D. T. Ferrughelli, E. Hellwege, J. E. Pond, W. J. Spond, J. Fowlks and our math clerk, S. Walther. We also wish to thank the personnel of the Mechanical Division who contributed to the program, in particular S. Pampinto, T. Sutowski, T. Morrison, D. J. Cecchini, R. A. Van Sweringen, T. E. Artz, F. D. Huber, E. E. Poole, T. J. Morgan and H. T. Oakley. In addition, we wish to acknowledge the help given by V. S. Engleman and C. A. Gagliardo in assisting in the preparation of the report. A special acknowlegement goes to M. Malinowsky who typed this report.

The personnel of the Industrial Environmental Research Laboratory of the EPA have been most helpful and deserve special thanks. We wish to express our gratitude for the help of D. B. Henschel, the EPA Project Officer, P. P. Turner and R. P. Hangebrauck.

# SECTION 1

## SUMMARY

The pressurized fluidized bed combustion of coal (PFBC) was studied in two experimental units, a continuous unit, also called the miniplant, and a smaller semi-batch unit. The study of the regeneration of sulfated $SO_2$ sorbent was also begun in the miniplant regenerator. The overall objectives of the experimental combustion program were to characterize the $SO_2$, $SO_3$, $NO_x$, CO and particulate emissions from a PFBC unit while varying both operating conditions and the coal and sorbent type, to measure combustion efficiency and heat transfer coefficients while varying operating conditions and preceding objectives. The objective of the regeneration program during this period was to shakedown the miniplant regenerator, developing procedures and equipment as needed. The shakedown was completed by a continuous combustion/regeneration run of 24 hours duration.

### MINIPLANT COMBUSTION STUDIES

The miniplant combustion section consists of a combustor vessel, refractory lined to an inside diameter of 32 cm (12.5 in). The overall height is 10 m (33 ft). A number of vertical water-cooled tubes are mounted in the combustor to remove the heat of combustion. Premixed coal and sorbent are injected into the combustor at a single point 28 cm (11 in) above the fluidized bed support grid. The combustor is capable of operating at pressures up to 1000 kPa (10 atm), at temperatures up to the ash agglomeration temperature of the coal (usually less than 980°C), at superficial velocities of up to 3 m/s (10 ft/sec) and with expanded beds of up to 6.1 m (20 ft). The maximum design coal feed rate is 220 kg/hr (480 lb/hr). Flue gas leaving the combustor passes through two cyclones in series to remove particulate matter. Particulates captured in the first cyclone are recycled to the combustor to improve combustion efficiency. Particulates captured in the second stage cyclone are rejected through a lock hopper. Spent sorbent is also rejected from the combustor through a lock hopper system to maintain a constant bed level in the combustor.

Runs were made with an Eastern bituminous Pittsburgh seam coal (Champion) containing 2% sulfur and screened to a particle size distribution of 200 to 2400 microns. Two sorbents were used: a Virginia limestone (Grove No. 1359) and an Ohio dolomite (Pfizer No. 1337). Both were screened to a size range of 840 to 2400 microns.

As of July 1976, the miniplant has accumulated over 1100 hours of coal combustion time in operations of up to 240 hrs. duration. Approximately 110 runs have been completed, 40 during the shakedown phase and 70 during the operating phase. Mechanical performance of the combustor was good. The coal feeding system performed satisfactorily. The vertical cooling coils installed to promote uniform temperatures in the combustor performed well after modifications were made to prevent distortion and erosion. Uniform combustor temperatures were achieved using the vertical coils with temperature differences usually less than 35°C (63°F) across the expanded bed.

The longest run duration was 240 hrs. This run, which was made in November 1975, completed the demonstration of the miniplant combustor. Continuous steam generation was maintained during the entire run while several coal feeding interruptions totaling 4 hrs. did occur. The unit was burning coal 98.5% of the time. During the last 101 hrs. of the run coal injection was uninterrupted.

$SO_2$ retention studies were made with dolomite No. 1337 over a range of operating conditions. The most important variable affecting $SO_2$ emissions was the Ca/S feed molar ratio (moles of calcium fed in the sorbent to moles of sulfur fed in the coal). The stoichiometric Ca/S ratio for the desulfurization reaction is 1.0. In the experiments reported here, the Ca/S ratio was varied from approximately 0.5 to 2.0, over which range the $SO_2$ retained by the dolomite bed varied from approximately 40 to 95%. A desulfurization reaction rate expression was developed which allowed the analysis of the effects on $SO_2$ retention of dolomite conversion (or utilization) levels and gas phase residence time. The effect of dolomite utilization on the reaction rate constant was developed using data obtained by a number of laboratories operating a variety of FBC experimental units of differing sizes and geometries. The agreement between results obtained in the various experimental units was good. Increasing dolomite utilization was found to decrease the reaction rate constant sharply. The effect of gas phase residence time, calculated as the ratio of the expanded bed height to the superficial gas velocity, was predicted using a first order reaction rate expression and the predicted results were verified reasonably well by a series of runs made over a residence time range of 0.8 to 2.8 s. The effect of residence time is more pronounced at higher $SO_2$ retention levels and the magnitude is such that, for 90% $SO_2$ retention, decreasing the residence time from 3 to 0.5 s would require doubling the Ca/S ratio from 1.5 to about 3.0. The effect of temperature on $SO_2$ retention was measured by varying the combustor temperature from 690 to 950°C (1270 to 1740°F). At temperatures between 840 and 950°C (1540 to 1740°F), no effect of temperature was observed. $SO_2$ retention levels measured at 690 to 760°C (1270 to 1400°F) were slightly lower than those measured at the higher temperatures. The average sorbent particle size was varied by a factor of two and no effect on $SO_2$ retention was observed. Pressure was varied from 600 to 930 kPa (6 to 9 atm abs.) and no effect was found. Variation in the excess air level from approximately 5 to 100% had no obvious effect on $SO_2$ retention.

$SO_2$ emissions were also measured with limestone No. 1359. Contrary to the results seen with dolomite sorbent, a marked effect of temperature was found, with increasing temperature giving higher $SO_2$ retention levels. The $SO_2$ retention levels were also lower than those observed using dolomite sorbent. These effects were due to the inability of the limestone to calcine completely under pressurized combustion conditions. Calcination greatly increases the porosity of the limestone, making the interior surface of the stone more accessible to the $SO_2$ reactant. At higher temperature conditions, e.g., 925-950°C (1700-1740°F) the limestone underwent extensive calcination, and although the limestone was not as active as dolomite, it was considerably more active than at the lower temperatures, i.e., 825-900°C (1520 to 1650°F), where the stone was largely in the carbonate form. At much lower temperatures, 670-760°C (1240-1400°F), the limestone was completely inactive. Pressure and sorbent particle size were found to have no effect on $SO_2$ retention.

Although an FBC utility boiler would normally be expected to operate in the temperature range of about 850 to 930°C (1550 to 1750°F), operation at temperatures down to about 700°C (1300°F) would be required to turn-down the boiler output to match a decrease in the electrical power demand. A series of runs was made using dolomite and limestone sorbent at temperatures near 700°C to determine the behavior of the FBC system at these lower temperatures. Some runs were also made at temperatures as low as 690°C (1270°F) to determine the lowest limit of operability. The minimum temperature at which combustion was stable was 690°C. An attempt to decrease the temperature to 600°C (1110°F) was unsuccessful. At that temperature, temperature control in the combustor became erratic and carbon monoxide emissions in the flue gas increased sharply, denoting poor combustion. The effect of the low temperature operation on $SO_2$ emission control was noted previously. A slight decrease in $SO_2$ retention was seen using dolomite sorbent at low temperatures. However, limestone was completely inactive and therefore, cannot be used in a pressurized FBC unit unless some means of increasing its activity under low temperature "turn-down" conditions can be found. Precalcination of the limestone is one possible way to do this and will be studied in the future.

As a result of the above studies, the sorbent requirements needed to satisfy the current EPA new source performance standards for $SO_2$ emissions from a coal fired boiler (1.2 lb $SO_2$/M BTU coal fired) can be estimated. This was done for coals containing 2 to 5% sulfur using the data obtained for the 2% sulfur coal and extrapolating to higher sulfur levels. Dolomite requirements were found to be less than those for limestone when expressed as the required Ca/S molar ratio. However, when expressed on a weight basis, e.g., wt. of sorbent/wt. of coal, limestone requirements were found to be slightly lower for coals containing up to 3% sulfur. For higher sulfur content coals, dolomite requirements were found to be less. For example, a coal containing 4% sulfur was estimated to require 34 kg limestone per 100 kg coal compared to 29 kg dolomite/100 kg coal. However, as cautioned previously, limestone may not be suitable for pressurized FBC applications unless precalcined limestone is found to be suitable for operation at the low temperature "turndown" conditions.

$SO_3$ emissions were found to be highly variable, averaging 6 ppm for one series of runs, and 23 ppm for another with individual runs showing even higher concentrations. No correlation was found with operating conditions and the cause for the formation and variability of $SO_3$ emissions is not understood. One possible explanation is sampling errors.

$NO_x$ emissions were measured and found to vary from 50 to 200 ppm or 0.04 to 0.17 g (as $NO_2$)/MJ (0.1 to 0.4 lb/M BTU). Although the operating conditions varied greatly, the only significant variables were excess air and combustor temperature. The $NO_x$ emissions increased four fold, from 0.04 to 0.17 g/MJ over a 5 to 110% range of excess air. The temperature effect in the 670 to 940°C (1240 to 1750°F) range was secondary and caused only a 25% increase in the emission level. The emissions are well below the EPA new source performance standard of 0.3 g/MJ (0.7 lb/M BTU) and have an average value of only 0.09 g/MJ (0.2 lb/M BTU) at 15% excess air, the level most likely to be used in a utility boiler.

CO emissions were low, generally in the range of 70 to 235 ppm at combustor temperatures above 825°C (1500°F). As the temperature was reduced below 825°C, CO emissions increased sharply to 300 to 800 ppm at 700 to 760°C (1300 to 1400°F).

Particulate emissions and sorbent losses by elutriation were also studied. Elutriation losses were found to increase with superficial gas velocity, with dolomite showing significantly higher loss rates. Limestone elutriation losses averaged about 10% of the limestone feed up to a superficial velocity of 2.1 m/s (7 ft/sec), and were equivalent to a feed Ca/S molar ratio of 0.24. Dolomite elutriation losses average about 40% of the feed, equivalent to a Ca/S molar feed ratio of 0.4. However, the losses were much higher at higher superficial velocities, reaching 110% of the dolomite feed rate at a velocity of 3 m/s (9.8 ft/sec), equivalent to a Ca/S feed rate of 0.8. This would indicate that the maximum superficial velocity possible with dolomite sorbent may be around 2.5 m/s (8 ft/sec).

Particulate concentrations in the flue gas leaving the second stage cyclone normally ranged from 0.9 to 4.8 g/m$^3$ (0.4 to 2.1 gr/SCF) with a mass median particle size of about 7 microns. The particulate captured in the second stage cyclone had a mass median particle size of about 20 microns.

Particulate composition data were also measured. The solid material removed from the combustor normally contained less than 1% unburned carbon, 2 to 25% coal ash and 75 to 95% used sorbent. The material captured by the second stage cyclone normally contained 3 to 20% unburned carbon, 40 to 80% coal ash and 15 to 60% used sorbent. These figures varied somewhat with the sorbent type. For example, the weight fraction of used sorbent in the second cyclone capture increased when dolomite was used, reflecting the higher dolomite elutriation loss rates. The fine particulates passing through the second stage cyclone contained 2 to 7% unburned carbon, 65 to 80% coal ash and 10 to 30% used sorbent.

Carbon combustion efficiency was found to increase with combustor temperature, reaching over 99% at a temperature of 940°C (1720°F). However, at lower temperatures, the combustion efficiency was found to vary considerably between 95 and 98%. No reason for the variation at the lower temperatures was found. Essentially all of the combustible carbon loss was due to carbon particulates removed in the second stage cyclone.

Heat transfer coefficients were found to vary from 250 to 420 W/m$^2$K (45 to 75 BTU/hr ft$^2$ °F), increasing with temperature and elevation in the bed and decreasing with sorbent particle size.

## BATCH UNIT COMBUSTION STUDIES

The smaller and older of the two pressurized FBC experimental units is the batch unit, so named because only coal is fed continuously to the combustor. The sorbent is added batch-wise to the combustor before a run and is removed from the combustor and analyzed after a run had been completed. The combustor consists of a vessel refractory lined to an inside diameter of 11.4 cm (4.5 in) with an overall height of 4.9 m (16 ft). Three vertical cooling coils are mounted in the combustor to remove the heat of combustion. Flue gas leaving the combustor passes through two cyclones and a final filter in series to remove particulate matter. Particulates captured in each removal device are removed after the completion of a run. Since the sorbent is not fed continuously to the combustor, the $SO_2$ emissions in the flue gas continually increase as the sorbent is sulfated. The $SO_2$ retention measured at the end of a run, together with the degree of calcium sulfation measured in the sorbent removed from the bed at the end of a run are used to calculate an equivalent Ca/S molar feed ratio. This permits the comparison of the desulfurization results measured in the batch unit and in continuous units such as the miniplant.

$SO_2$ emissions were measured using an Eastern bituminous Pittsburgh seam coal (Arkwright), containing 2.6% sulfur, an Illinois No. 6 bituminous coal containing 4% sulfur, and a Western subbituminous coal containing 0.7% sulfur. Grove No. 1359 limestone and an Ohio dolomite (Tymochtee) were the sorbents. The $SO_2$ retention data obtained with the Eastern coal and dolomite sorbent agreed fairly well with data obtained in the miniplant after correcting for differences in the gas phase residence times, and considering the inherent differences between a batch and continuous system. However, limestone desulfurization results measured in the batch unit exhibited a fair degree of data scatter and were difficult to interpret. For example, data obtained with the Eastern coal showed no effect of temperature, contrary to the results obtained in the miniplant. Runs with Illinois coal showed a temperature effect, but $SO_2$ retention levels were much lower than expected.

Runs made with the low sulfur Western coal using limestone sorbent gave very low $SO_2$ emissions. This indicated the possibility of significant $SO_2$ retention by the high calcium content of the coal ash. Runs were then made with inert bed material in place of the limestone and $SO_2$ retention levels on the flyash of about 50% were measured.

$NO_x$ emissions were also measured in the batch unit. The emissions increased with increasing excess air. In the range of 15 to 20% excess air, the emissions averaged 0.09 to 0.13 g/MJ, (0.2 to 0.3 lb/M BTU), slightly higher than the level measured in the miniplant. However, at higher excess air levels, the $NO_x$ emissions were significantly higher than those measured in the miniplant. Runs made with the Western coal at excess air levels of over 150%, indicated $NO_x$ emissions could be as high as 0.4 g/MJ (0.9 lb/M BTU). Further studies with Western coal will be made in a continuous unit to determine if the $NO_x$ emissions are higher than those obtained with Eastern and Illinois coals.

CO emissions measured in the batch unit averaged about 180 ppm at higher temperatures, within the range measured in the miniplant.

Particulate emissions from the combustor and the cyclones were measured by weighing the particulate matter captured in the cyclones and filter. Emissions from the combustor averaged 16 $g/m^3$ (7 gr/SCF), 2 $g/m^3$ (0.9 gr/SCF) from the first cyclone and 1 $g/m^3$ (0.5 gr/SCF) from the second cyclone.

Carbon combustion efficiencies increased with temperature and excess air, exceeding 98% at excess air levels of 60% or more and temperatures generally in excess of 900°C (1650°F). At 20% excess air, the combustion efficiency was about 93% at 850 to 870°C (1560-1600°F) and 97% at temperatures above 900°C (1650°F). Combustion efficiencies were generally lower than those measured in the miniplant, probably due to the lack of recycle of first stage cyclone particulates in the batch unit.

## MINIPLANT REGENERATOR SHAKEDOWN

The regenerator consists of a refractory lined vessel with an inside diameter of 22 cm (8.5 in) and an overall height of 6.7 m (22 ft). Gaseous fuel is burned in a plenum below the fluidized bed to achieve the reaction temperature. Additional fuel is injected directly into the fluidized bed just above the fluidizing grid to create a reducing zone in which the $CaSO_4$ reduction reaction occurs. Supplementary air is injected directly into the bed at a higher elevation to create an oxidizing zone. The oxidizing environment at the top of the bed assures high selectivity to CaO, the desired product of the regeneration reaction, by minimizing the formation of CaS, an undesired by-product.

The shakedown of the regenerator proceeded in two phases. In the first, the regenerator was operated in a batch fashion, uncoupled from the combustor, to check out the system and develop necessary equipment and procedures. In the second phase, provisions for the continuous transfer of solids between the combustor and regenerator were developed and the regenerator, coupled to the combustor, was operated continuously for a period of 24 hrs.

In the batch operating mode, procedures were developed which gave good control of the bed temperature, good temperature distribution and prevented the agglomeration of the bed by temperature excursions. This was done primarily by proper adjustment and balance of the fuel and air flows into the system and by the design of a fluidizing grid which promoted solids mixing.

$SO_2$ levels measured in the regenerator off gas during the batch tests were 3% or lower. These concentrations were only about 50% of the concentrations calculated assuming equilibrium was established between the solids and the gas. However, the $SO_2$ concentrations measured in the batch runs agreed closely with concentrations obtained experimentally by another investigator (10) under equilibrium conditions indicating the calculated concentrations may not be correct. Conversion of the sulfated sorbents was over 90% in the batch tests and selectivity to CaO was over 99%.

The second shakedown phase began by developing a system to transfer solids continuously between the combustor and regenerator vessels. The system consisted in setting the pressure in the regenerator very slightly higher than the combustor and using the pressure differential and a pulsing flow of nitrogen to move the solids in a controlled fashion between the vessels. A transfer line lock hopper was used to move the solids from the combustor to regenerator. Two water cooled knife valves were designed and built for this service. The valves were opened and closed by a timing mechanism in such a way as to permit solids to flow by gravity from the combustor into the transfer line. The transfer line was then isolated, increased slightly in pressure and then drained into the regenerator. This system performed satisfactorily during checkout and ran without failure during the 24 hr. shakedown run. In this run, limestone was added to the combustor in an amount required to make up for entrainment losses. $SO_2$ emissions from the combustor were under 400 ppm and $SO_2$ in the regenerator off gas was between 0.7 and 1.0%. This $SO_2$ concentration was limited not by chemical equilibrium or reaction rates, but rather by the volume of gas resulting from the fuel which had to be burned to maintain the regenerator at reaction temperatures. It is not indicative of the $SO_2$ level which can be reached in a larger system with lower heat losses. As the limestone was cycled between the combustor and regenerator, attrition and entrainment losses decreased and no limestone was fed to the combustor for the last 10 hrs. of the test.

## CONTINUING STUDIES

Additional combustion studies will be carried out in the miniplant in which an Illinois No. 6 coal containing approximately 4% sulfur will be burned. Precalcined limestone sorbent will also be used in another series of tests to determine its activity at low "turndown" temperatures. The batch combustion unit will be converted to a continuous unit and used to support the miniplant program.

A particulate control program will be started. A granular bed filter will be installed on the miniplant flue gas stream and shakedown will begin. The purpose of the program will be to determine if this type particulate control device can satisfy environmental and gas turbine particulate requirements. It will also be used in a long term materials testing program sponsored by the Energy Research and Development Administration (ERDA).

A program aimed at making a comprehensive analysis of all potentially harmful emissions from a pressurized fluidized bed combustion system will begin. This will be done as part of the overall EPA FBC environmental assessment program.

Regeneration studies will continue. The first goal will be to conduct a 100 hr run demonstrating the feasibility of the continuous FBC combustion and sorbent regeneration concept. This will be followed by an optimization study aimed at measuring $SO_2$ content in the regenerator off gas and regenerated sorbent activity maintenance.

## SECTION II

## INTRODUCTION

The pressurized fluidized bed combustion of coal is a new combustion technique which can reduce the emission of $SO_2$ and $NO_x$ from the burning of sulfur-containing coals to levels meeting EPA emission standards. This is done by using a suitable $SO_2$ sorbent such as limestone or dolomite as the fluidized bed material. In addition to emissions control, this technique has other potential advantages over conventional coal combustion systems which could result in a more efficient and less costly method of electric power generation. By immersing steam generating surfaces in the fluidized bed, the bed temperature can be maintained at low and uniform temperatures in the vicinity of 800 to 950°C. The lower temperatures allow the use of lower grade coals (since these temperatures are lower than ash slagging temperatures), and also decrease $NO_x$ emissions. Operation at elevated pressures, in the range of 600 to 1000 kPa, offers further advantages. The hot flue gas from a pressurized system can be expanded through a gas turbine, thereby increasing the power generating efficiency. Operation at the higher pressure also results in a further decrease in $NO_x$ emissions.

In the fluidized bed boiler, limestone or dolomite is calcined and reacts with $SO_2$ and oxygen in the flue gas to form $CaSO_4$ as shown in reaction (1).

$$CaO + SO_2 + 1/2O_2 \rightarrow CaSO_4 \qquad (1)$$

Fresh limestone or dolomite sorbent feed rates to the boiler can be reduced by regeneration of the sulfated sorbent to CaO and recycle of the regenerated sorbent back to the combustor. One regeneration system, studied by Exxon Research and Engineering Company in the past, is the so-called one step regeneration process in which sulfated sorbent is reduced to CaO in a separate vessel at a temperature of about 1100°C according to equation (2). $SO_2$ in the regenerator off gas is at a sufficiently high concentration to be recovered in a by-product sulfur plant.

$$CaSO_4 + \overset{CO}{H_2} \rightarrow CaO + SO_2 + \overset{CO_2}{H_2O} \qquad (2)$$

A diagram of the pressurized fluidized bed combustion and regeneration process is shown in Figure II-1.

Exxon Research and Engineering Company, under contract to the EPA, has built two pressurized fluidized bed combustion units to study the combustion and regeneration processes. The smaller of the two units, the batch unit, was built under contract CPA 70-19 and was described in previous reports (1)(2). Figure II-2 is a photograph of the batch units. Those reports described regeneration and combustion studies carried on in the batch unit. The subsequent coal combustion studies carried out in the batch unit under Contract 68-02-1451 are described in this report. The program was aimed at the development of equipment and operating techniques, the study of the effect of process conditions on $SO_2$, $NO_x$ and CO emissions, the measurement

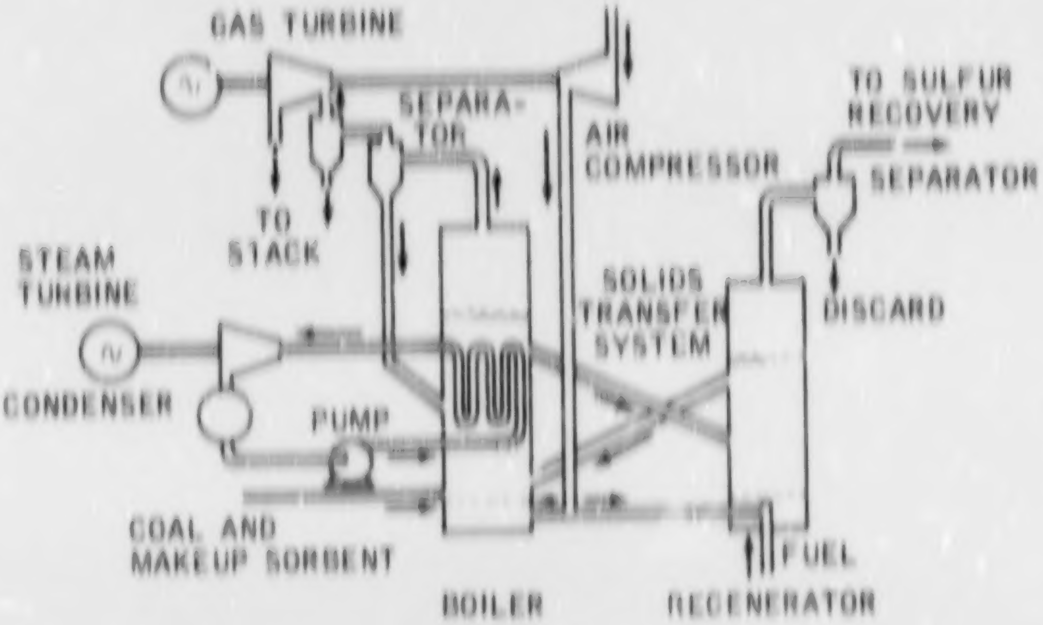
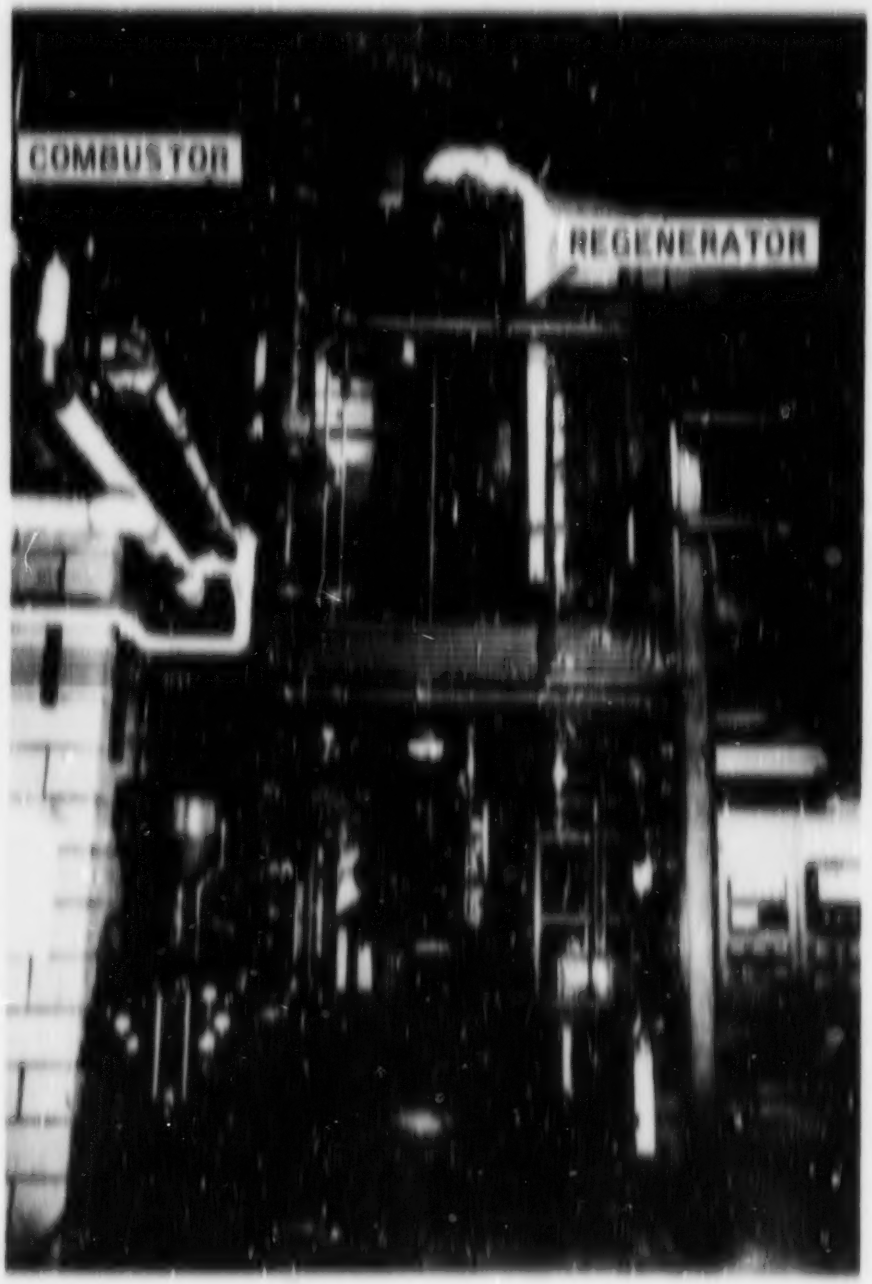

GAS TURBINE
SEPARATOR
TO SULFUR RECOVERY
AIR COMPRESSOR
SEPARATOR
TO STACK
STEAM TURBINE
SOLIDS TRANSFER SYSTEM
DISCARD
CONDENSER
PUMP
COAL AND MAKEUP SORBENT
FUEL
BOILER
REGENERATOR

FIGURE II-1

PRESSURIZED FLUIDIZED BED COAL COMBUSTION SYSTEM

FIGURE II-2

BATCH FLUIDIZED BED COAL COMBUSTION UNIT


COMBUSTOR
REGENERATOR

of combustion efficiency, particulate emissions and the measurement of heat transfer coefficients between the fluidized bed and steam tubes. Various coals and sorbents were also tested.

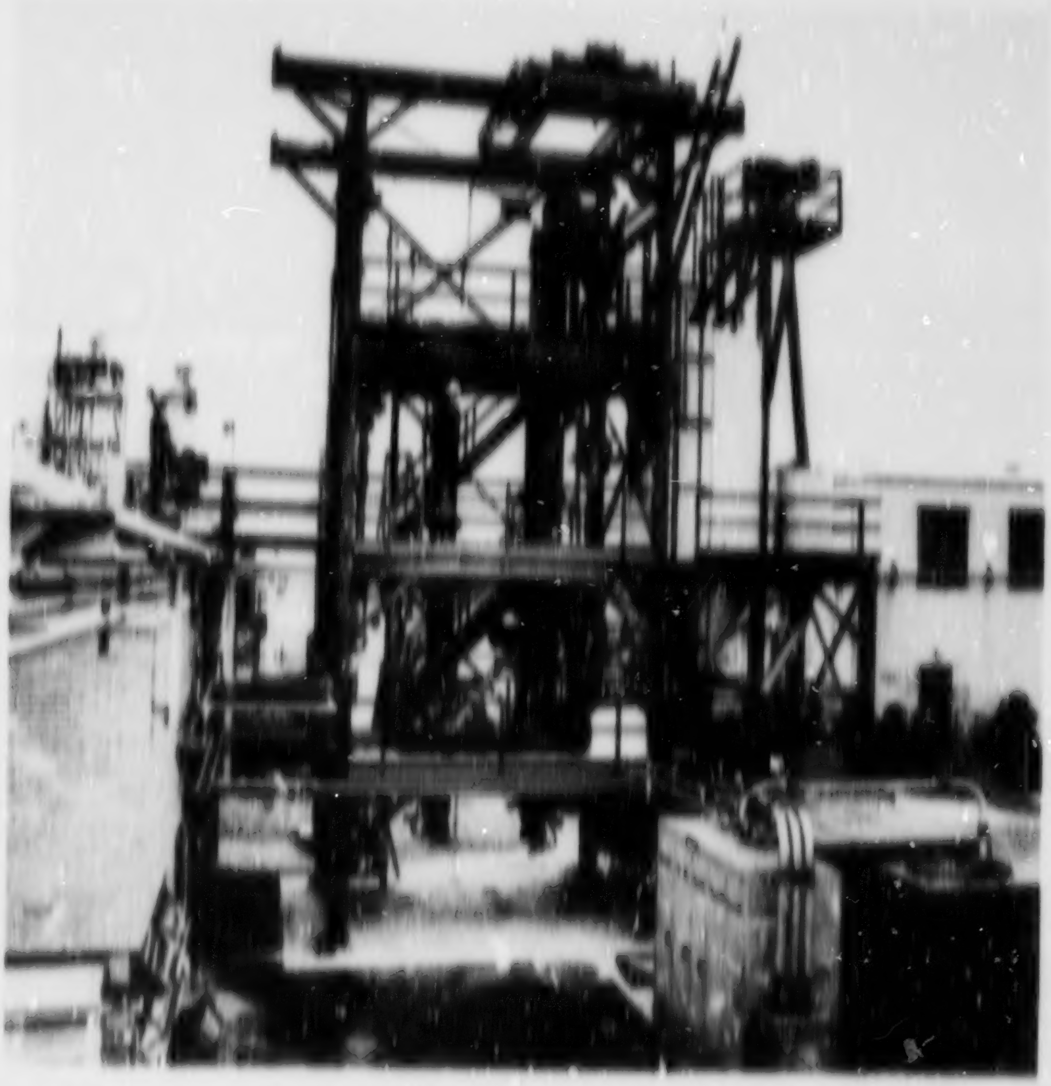
The larger unit, called the miniplant, was designed under EPA Contract CPA 70-19 and built under Contract 68-02-0617. Figure II-1 shows a photograph of the miniplant. The shakedown and operation of the unit was funded under Contract 68-02-1312. Previous reports (1)(2) described the design, shakedown and initial operation of the unit. This report includes additional results from the operation of the combustion section of the miniplant. The effect of operating conditions on $SO_2$, $NO_x$, CO and particulate emissions, on combustion efficiency, sorbent attrition and heat transfer coefficient was measured. Various sorbents were tested.

This report also describes the regeneration section of the miniplant and the initial results of the regenerator shakedown activity.

The period of performance discussed in this report is July 1, 1975 to July 30, 1976.

FIGURE II-3

EXXON FLUIDIZED BED COMBUSTION MINIPLANT



## SECTION III

## COMBUSTION STUDIES

Combustion studies have been carried out in two pressurized fluidized bed units. The smaller and older of the two units is the batch unit. This unit is capable of continuous coal addition, but does not have provisions for continuous sorbent addition or continuous removal of sulfated sorbent from the fluidized bed. The miniplant is the larger of the two units and has provisions for continuous addition of coal, and sorbent and continuous withdrawal of sulfated sorbent. This section of the report describes combustion studies carried out in both experimental units.

### MINIPLANT COMBUSTOR

The miniplant is shown schematically in Figure III-1. As of July 1976, the combustor has been operated for a total of approximately 1100 hours in a series of individual runs of up to 240 hours duration. This section of the report describes the combustor equipment, operating procedures, combustor performance and combustion results. A discussion of the regenerator section is given in Section IV.

#### Equipment, Materials, Procedures

This section will focus on the major system components which include: 1) solids feeding system, 2) combustor with internal subcomponents, 3) combustor cyclones, 4) pressure control and flue gas discharge system, 5) flue gas sampling and analytical system, 6) process monitoring and data generation system, and 7) combustor safety and alarm system. A detailed description of each of these systems can be found in an earlier report and only a brief discussion will be included here (2).

Figure III-2 shows a schematic of the miniplant coal and sorbent feeding system. Solids are fed under atmospheric pressure from storage bins through a feeder and blender into a feed vessel. The coal/sorbent mixture is held in this vessel until refill of the injector vessel becomes necessary. The injector vessel is maintained at a pressure slightly higher than the combustor. Solids are fed from the bottom of the injector vessel through an orifice into the transport line. Transport air is then used to inject the solids into the combustor through an air-cooled nozzle. The solids feed rate is controlled by controlling the pressure difference between the injector vessel and combustor. The higher the pressure difference, the greater the solids feed rate, as long as the transport air rate is held constant. The solids feed rate is adjusted by a cascade control system which maintains a constant temperature in the combustor at a point close to the solids entry port. This control system continually readjusts the pressure differential between the injector vessel and the combustor to vary the coal injection rate in such a way as to maintain constant temperature in the combustor.

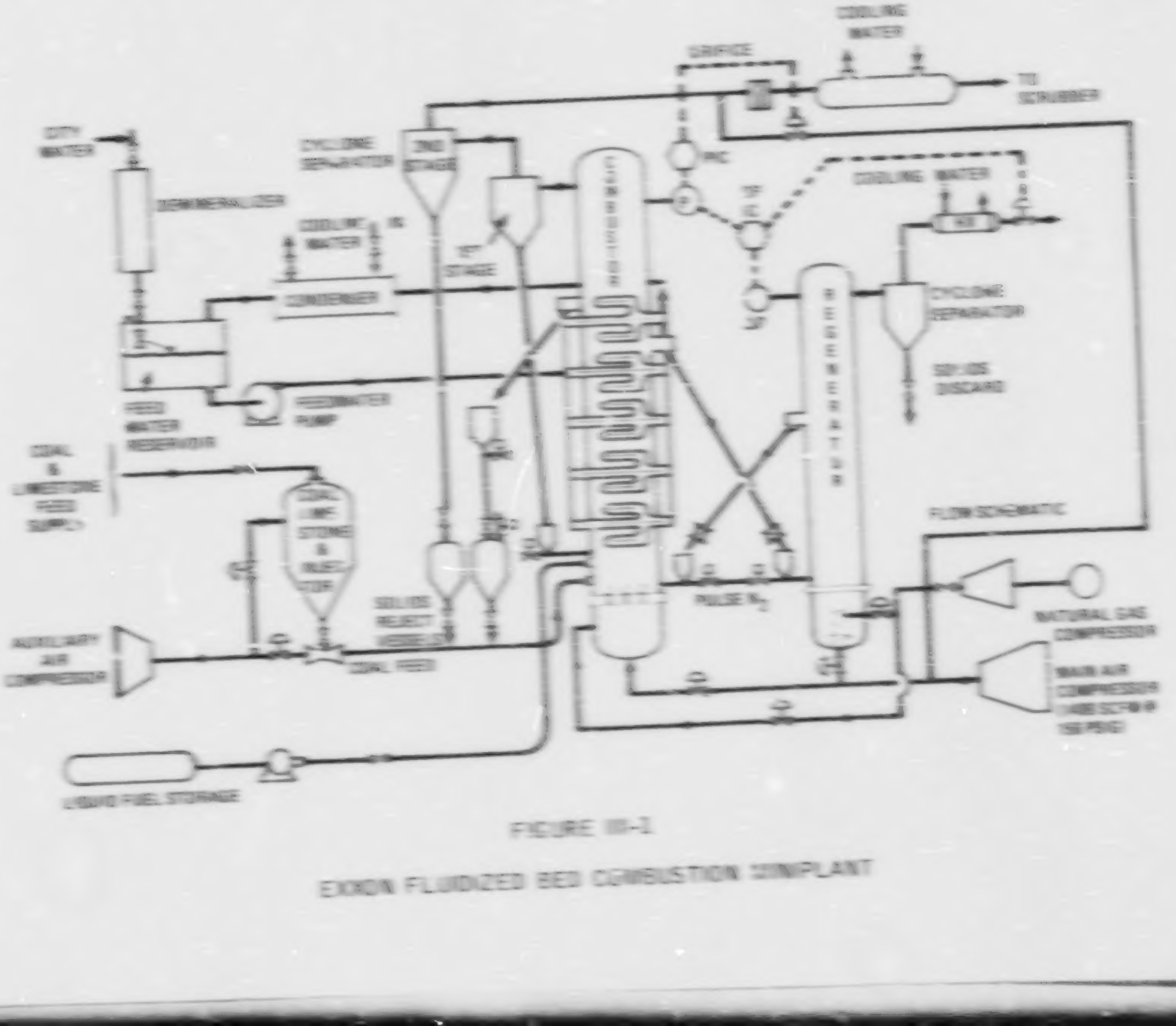
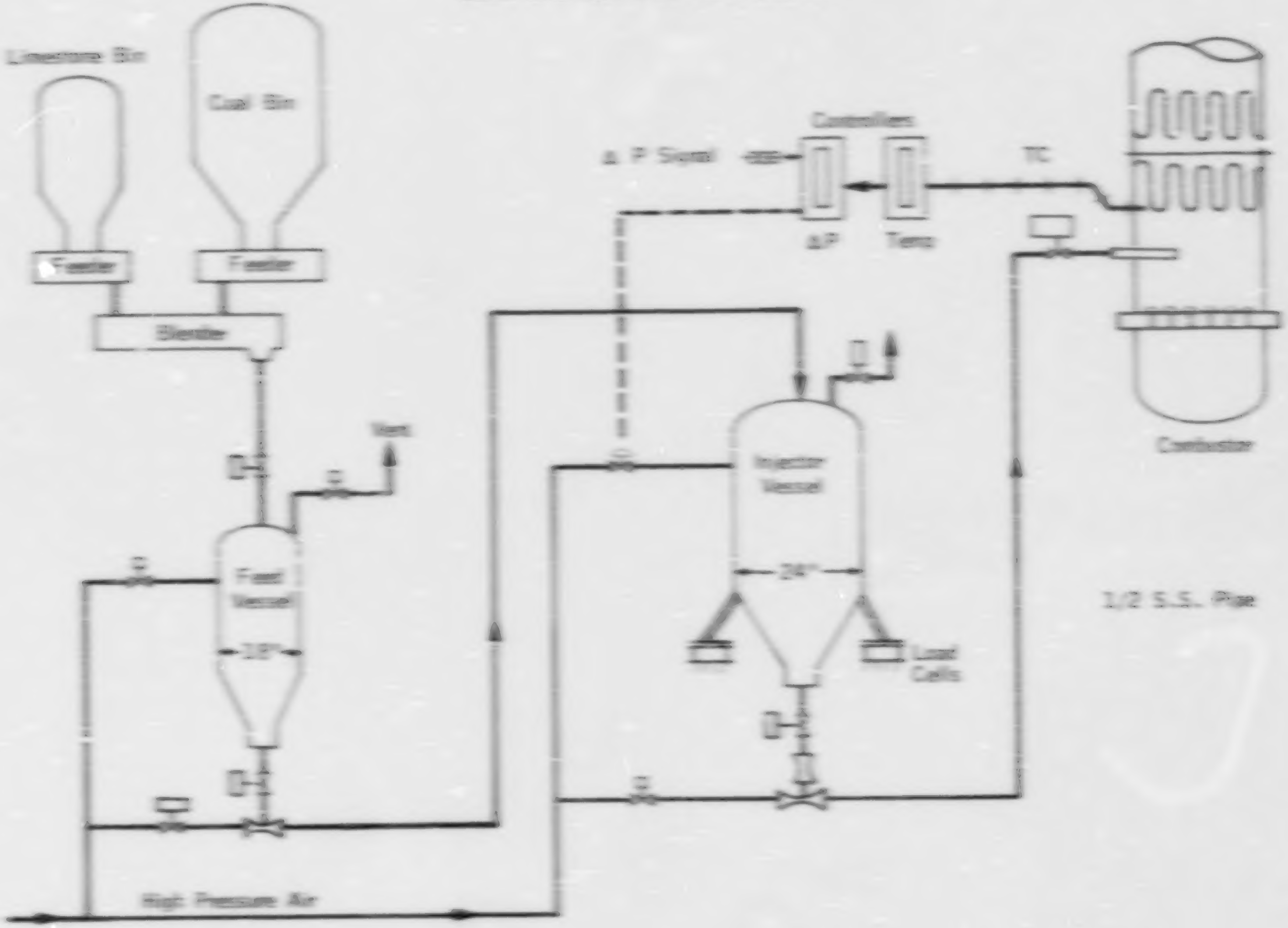

CITY WATER
DEMINERALIZER
CYCLONE SEPARATOR
2ND STAGE
1ST STAGE
COOLING WATER IN
CONDENSER
FEED WATER RESERVOIR
FEEDWATER PUMP
COAL & LIMESTONE FEED SUPPLY
COAL LIMESTONE & REJECT
SOLIDS REJECT VESSELS
AUXILIARY AIR COMPRESSOR
COAL FEED
LIQUID FUEL STORAGE
COMBUSTOR
ORIFICE
PIC
TIC
COOLING WATER
TO SCRUBBER
COOLING WATER
HX
REGENERATOR
CYCLONE SEPARATOR
SOLIDS DISCARD
FLOW SCHEMATIC
PULSE $N_2$
NATURAL GAS COMPRESSOR
MAIN AIR COMPRESSOR (1400 SCFM @ 150 PSIG)

FIGURE III-1

EXXON FLUIDIZED BED COMBUSTION MINIPLANT

## FIGURE III-2

## COAL & LIMESTONE FEED SYSTEM


Limestone Bin
Coal Bin
Feeder
Feeder
Blender
Vent
Feed Vessel
18"
Δ P Signal
Controllers
ΔP
Temp
TC
Injector Vessel
24"
Load Cells
Combustor
1/2 S.S. Pipe
High Pressure Air

The combustor consists of a 61 cm I.D. steel shell refractory lined to an actual internal diameter of 31.8 cm. The 9.75 m high unit is designed in flanged sections and contains various ports to allow for material entry and discharge. Numerous taps are also provided for monitoring both pressure and temperature. A schematic of the combustor is shown in Figure III-3. Combustion air to the unit is provided by a main air compressor having a capacity 40 stnd. $m^3$/min at 1030 kPa gauge (1400 scfm at 150 psig). The bottom plenum section houses the natural gas burner used for initial pre-heating of the bed during unit start-up. Fuel to the burner is provided by a natural gas compressor with a capacity of 0.57 stnd. $m^3$/min at 1379 kPa gauge (20 scfm at 200 psig). Once the fluidized bed temperature reaches 430°C, a liquid fuel system is used to heat the bed to the coal ignition temperature.

Heat removal from the combustor is provided by cooling coils located in discrete vertical zones above the grid. Each coil has a total surface area of 0.55 $m^2$ and consists of vertically-oriented loops constructed of 1.3 cm Schedule 40 316 stainless steel pipe. A closed-loop arrangement is used in which a feedwater reservoir provides the supply of demineralized cooling water. A high pressure pump is used to pump the water through the coils. The flow rate and exit temperature from each coil can be separately controlled and monitored. The steam-water mixture that exits from each coil is condensed, returned to the feedwater reservoir, and recirculated.

The combustion gases that exit the combustor go through a two-stage cyclone system. The primary intent of the first cyclone is to recirculate larger unburned carbon particles back to the combustor to improve combustion efficiency. Particles trapped by the secondary cyclone are dropped into a lockhopper and disposed of on a batch basis.

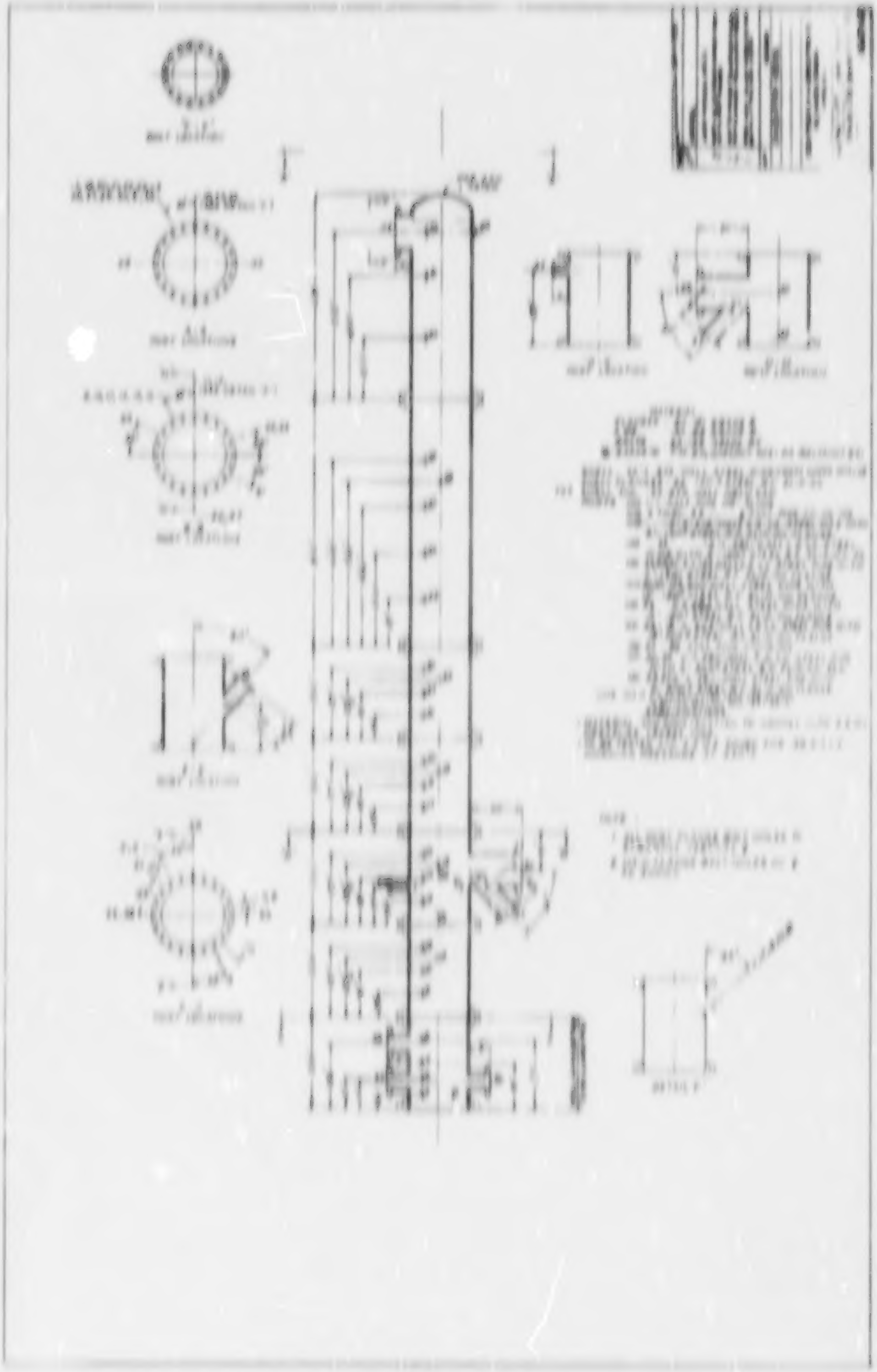
The technique used to control combustor pressure consists of allowing the system pressure drop to occur across an appropriately sized silicon carbide nozzle located in the flue gas exit line. Back pressure is controlled by regulating the flow of a secondary air stream to the nozzle inlet. After passing through the nozzle, the gases are cooled and then enter a scrubber for final cleanup before being discharged into the atmosphere.

Flue gas is sampled at a point about 7 m downstream of the second stage cyclone. The system is designed to produce a solids-free, dry stream of flue gas at approximately ambient temperature and atmospheric pressure whose gas composition is essentially unaltered from that of the original flue gas. A schematic is shown in Figure III-4. A flue gas particulate sampling system was also installed and is shown schematically in Figure III-5.

Solids rejection from the combustor is required to maintain a steady bed height when a mixture of coal and sorbent is fed. Solids are rejected through a port located 230 cm above the fluidizing grid. Solids flow by gravity through a refractory lined pipe into a "pulse pot" from where they are pneumatically transported by controlled nitrogen pulses to a pressurized lockhopper.

FIGURE III-3

COMBUSTOR VESSEL



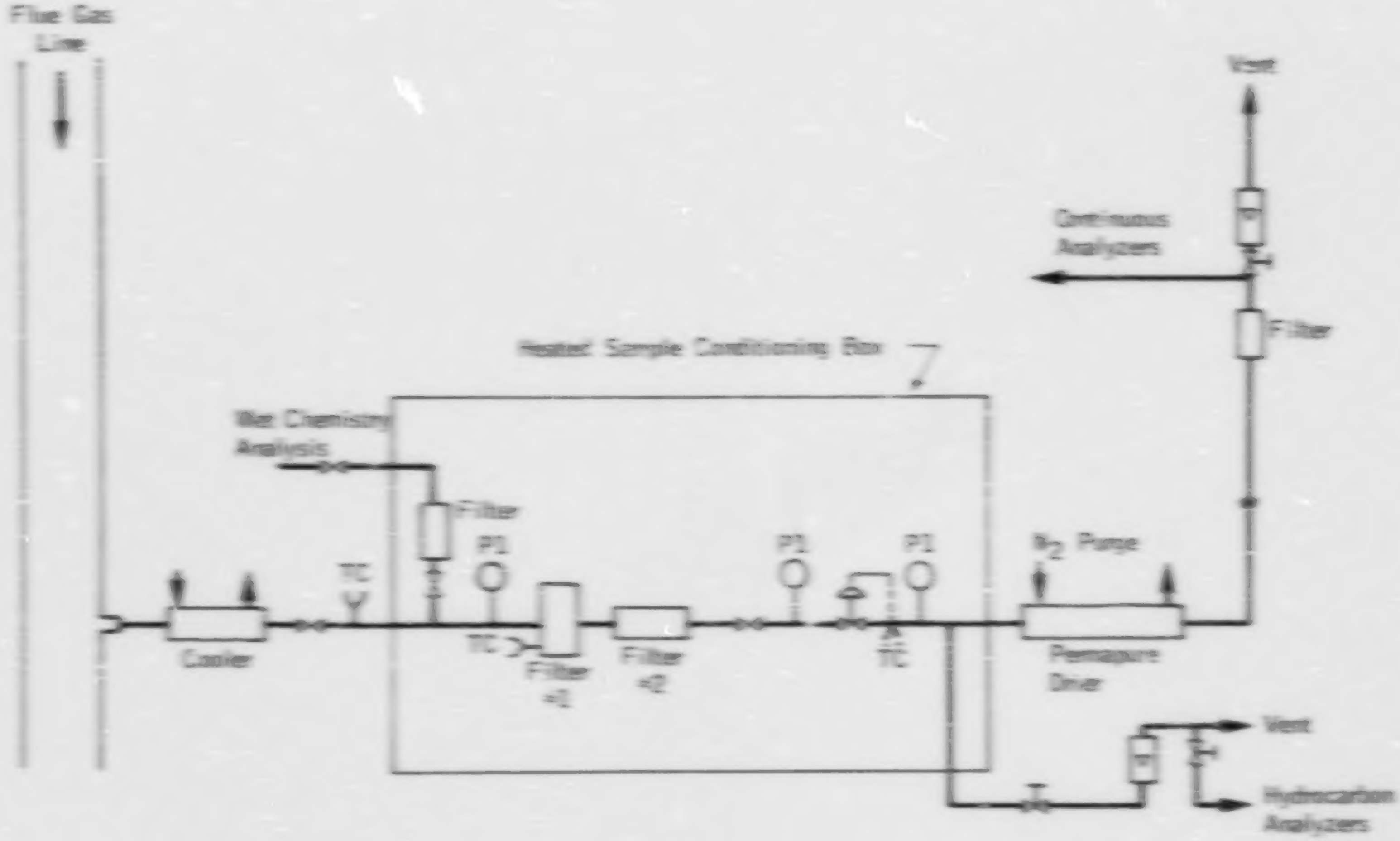

Flue Gas
Line
Cooler
TC
Wet Chemistry
Analysis
Filter
PI
TC
Filter
#1
Filter
#2
PI
PI
TC
Heated Sample Conditioning Box
$N_2$ Purge
Permapure
Drier
Filter
Continuous
Analyzers
Vent
Vent
Hydrocarbon
Analyzers

FIGURE III-4

FLUE GAS SAMPLING SYSTEM

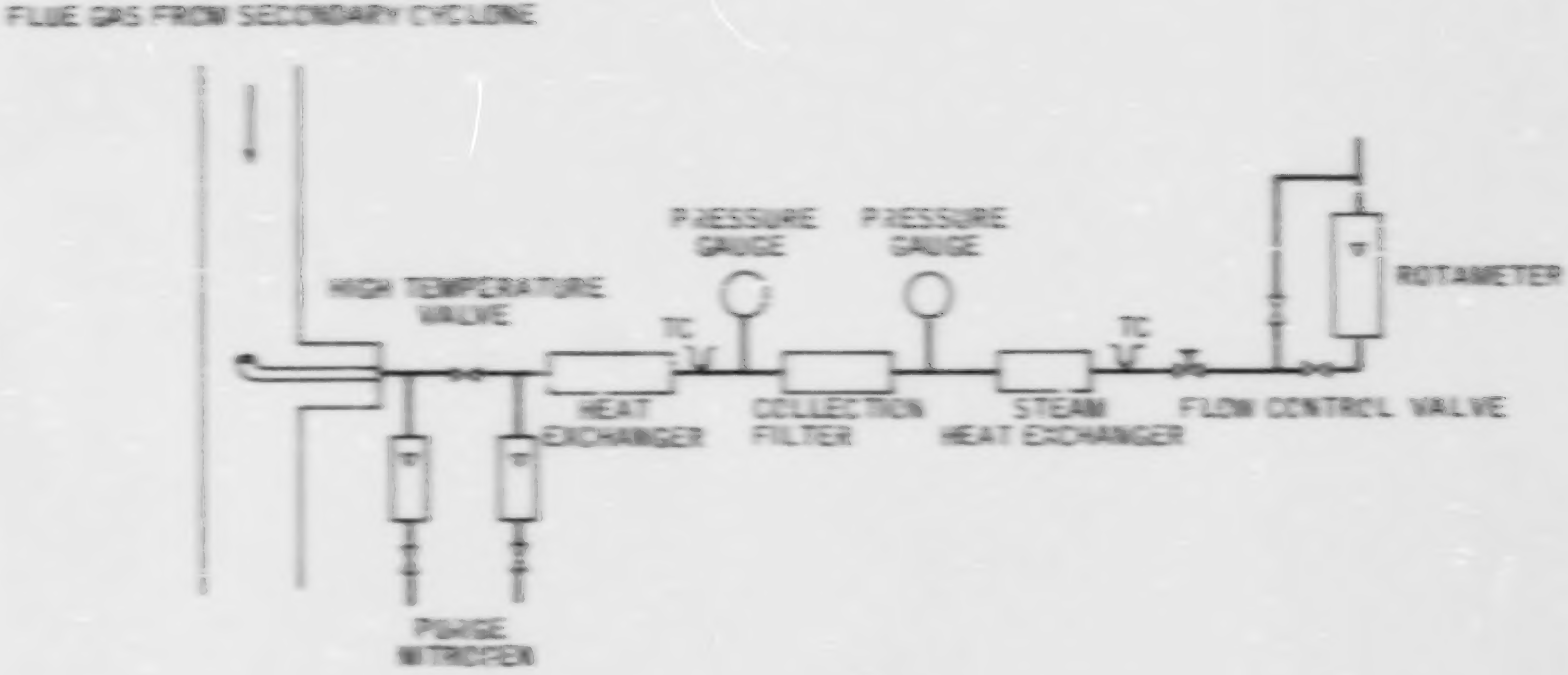

FLUE GAS FROM SECONDARY CYCLONE
HIGH TEMPERATURE VALVE
PURGE NITROGEN
HEAT EXCHANGER
TC
PRESSURE GAUGE
COLLECTION FILTER
PRESSURE GAUGE
STEAM HEAT EXCHANGER
TC
FLOW CONTROL VALVE
ROTAMETER

FIGURE III-5

MINIPLANT PARTICULATE SAMPLING SYSTEM

Data characterizing total system operation are recorded on three multipoint recorders. In addition, at one minute intervals, the same data are recorded by a data logger system consisting of a Digitrend 210 data logger with printer and a Kennedy 1701 magnetic tape recorder. The magnetic tape, containing about 3600 items of data per hour of run time, is fed to a computer which converts the data logger output signals to flow rates, pressures, etc. The data are then averaged and standard deviations calculated over pre-selected time intervals.

The unit is equipped with a process alarm system which was designed to warn of impending operational problems. Two general alarm categories exist. The first, dealing with less critical situations, alerts the operator of the problem so that appropriate corrective action can be taken. The second class of more critical alarms results in the immediate or time delayed shutdown of the system.

### Materials--

Coal--Coal used in the miniplant variables study was a high volatile bituminous coal obtained from the Consolidation Coal Company's "Champion" preparation plant in Pennsylvania. The coal is a Pittsburgh No. 8 seam coal and its analysis is shown in Table III-1. Coal samples were periodically sent to Exxon's Baytown, Texas, coal research facilities or to the Conoco Research and Development Company to assure accurate analysis. Grinding and sizing was done by the Penn-Rillton Company. Essentially all of the coal was less than 2380 um (No. 8 U.S. Mesh). Fines smaller than 40 mesh were partly removed. Two typical distributions are shown in Figure III-6.

Sorbents--Grove limestone (No. 1359) and Pfizer dolomite (No. 1337) were the primary sorbents used in the miniplant variables study. One run using Tymochtee dolomite was also made. The composition of these stone is given in Table III-2. Most of the runs were made with the stone screened to give a distribution with a minimum of 90% between 2380 um (No. 8 U.S. Mesh) and 841 um (No. 20 U.S. Mesh). A typical size distribution of the limestone is shown in Figure III-7. Runs to evaluate the effect of sorbent particle size on desulfurization were also made. For these runs particle sizes of 8 x 14 and 14 x 25 mesh were used.

### Operating Procedures--

Prior to initiating a run, a detailed checkout procedure is followed to insure that the system is ready for operation. This includes various equipment checks, calibration of flue gas analyzers, activation of process monitoring and control systems, and the turning on of all cooling water systems. All runs were begun with an initial bed of sorbent in the combustor. This consisted of either a fresh charge of uncalcined limestone or the bed from the previous run.

The first operation of startup involves heating the sorbent bed by burning natural gas in the burner plenum followed by injection of kerosene into the bed. Prior to ignition of natural gas, an air flow of about 9.9 stnd. $m^3$/min (350 scfm) or about half that used at normal operating conditions is fed through the burner while combustor pressure is raised to 280 kPa gauge. Once ignition begins, this procedure maximizes incoming gas temperature under conditions which allow good natural gas combustion and adequate bed fluidization. Water flow rates through the combustor cooling coils are kept low to reduce heat loss to the coils. Ignition begins by

TABLE III-1. MINIPLANT COAL ANALYSES

| Coal<br>Source of Analyses<br>Run No. | Component Weight Percent<br>Champion<br>Exxon<br>19.4-19.9 | <br>Champion<br>Exxon<br>20.1-27.3 | <br>Champion<br>Conoco R&D<br>20.1-27.3 | <br>Champion<br>Exxon<br>27.4-27.12 | <br>Champion<br>Conoco R&D<br>27.4-27.12 |
|---|---|---|---|---|---|
| Moisture | 2.21 | 2.52 | 2.37 | Dry Basis | 2.95 |
| Ash | 8.85 | 9.64 | 6.75 | 9.52 | 7.91 |
| Total Carbon | 78.16 | 75.31 | 72.69 | 75.26 | 73.34 |
| Hydrogen | 5.09 | 5.39 | 5.19 | 5.13 | 5.17 |
| Sulfur | 2.21 | 1.95 | 1.95-1.98 | 2.08 | 2.17-2.25 |
| Nitrogen | 1.61 | 1.57 | 1.52 | 1.62 | 1.50 |
| Oxygen (by difference) | 4.12 | 6.15 | | 6.39 | |
| Chlorine | 0.08 | 0.07 | N.D.[1] | 0.06 | N.D. |
| Higher Heating Value (BTU/lb) | 13,648 | 13,513 | N.D. | 13,577 | N.D. |

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(1) Not Determined

TABLE III-1 (continued). MINIPLANT COAL ANALYSES

|  | Component Weight Percent |  |  |  |  |
|---|---|---|---|---|---|
| Coal | Champion | Champion | Champion | Champion | Champion |
| Source of Analyses | Exxon | Conoco R&D | Exxon | Conoco R&D | Exxon |
| Run No. | 27.13-27.21,28 | 27.13-27.21,28 | 29-35 | 29-35 | 36.1 |
| Moisture | Dry Basis | 2.60 | Dry Basis | 2.73 | Dry Basis |
| Ash | 8.27 | 8.94 | 9.12 | 9.35 | 8.26 |
| Total Carbon | 75.83 | 72.66 | 75.64 | 72.29 | 72.43 |
| Hydrogen | 5.11 | 5.10 | 5.09 | 5.16 | 5.16 |
| Sulfur | 2.14 | 1.90-2.01 | 1.78 | 2.10-2.19 | 2.07 |
| Nitrogen | 1.60 | 1.49 | 1.62 | 1.46 | 1.40 |
| Oxygen (by difference) | 7.06 |  | 6.87 |  | 10.73 |
| Chlorine | 0.06 | N.D. | 0.06 | N.D. | 0.09 |
| Higher Heating Value (BTU/lb) | 13,711 | N.D. | 13,619 | N.D. | 13,735 |

FIGURE III-6

COAL PARTICLE SIZE DISTRIBUTION

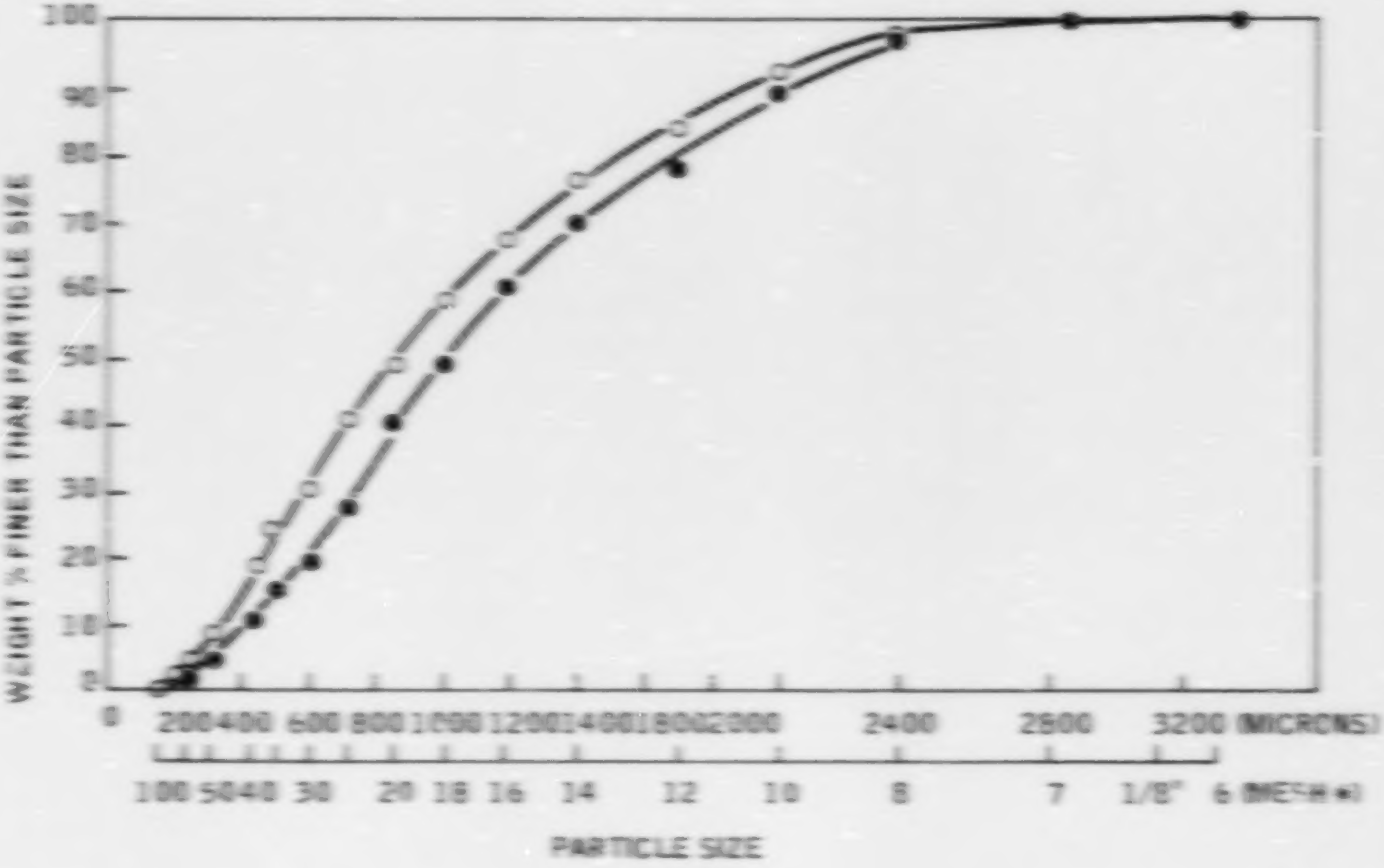
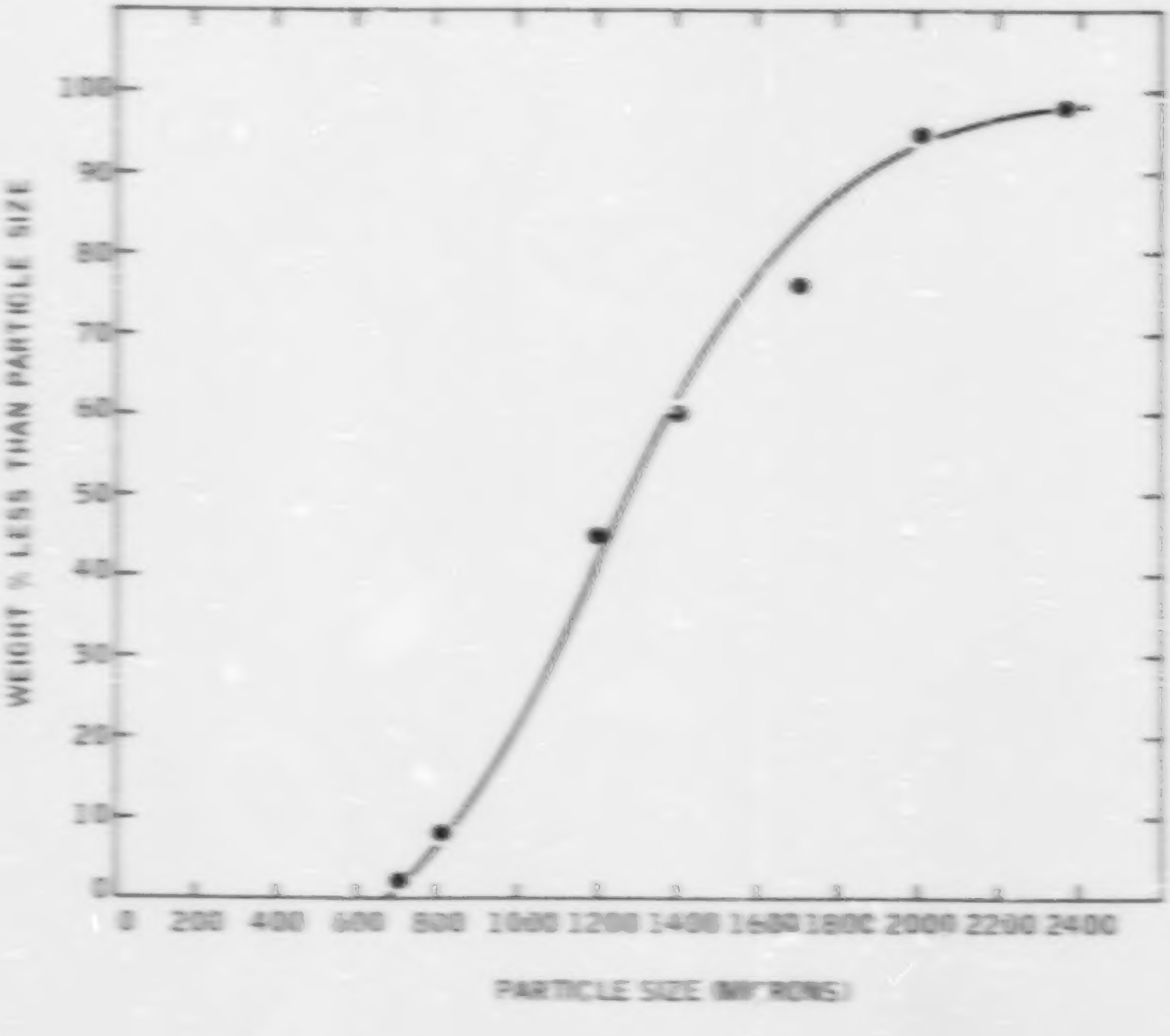


TABLE III-2. PROPERTIES OF SORBENTS USED
IN MINIPLANT VARIABLES STUDY

| Designation | Quarry Source | Stone Type | Chemical Analysis, wt % | | | | |
|---|---|---|---|---|---|---|---|
| | | | CaO | MgO | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ |
| 1359 | Grove Lime Co.<br>(Stephens City, VA) | Limestone | 97.0 | 1.2 | 1.1 | 0.3 | 0.2 |
| 1337 | Chas. Pfizer Co.<br>(Gibsonburg, OH) | Dolomite | 54.0 | 44.0 | 0.9 | 0.2 | 0.3 |
| Tymochtee | C. E. Duff and Sons<br>(Huntersville, OH) | Dolomite | 53.8 | 38.7 | 5.3 | 0.9 | 1.2 |

FIGURE III-7

LIMESTONE NO. 1359 PARTICLE SIZE DISTRIBUTION



simultaneously feeding 0.57 stnd. $m^3$/min (20 scfm) of natural gas through the burner while activating an ignition electrode.

Because of the limited capacity of the gas compressor, natural gas burned in the plenum is used only to heat the bed to a temperature of about 430°C, sufficient to insure self-ignition of kerosene. This generally requires 20-30 minutes. At this point, kerosene is injected into the lower portion of the bed. When rising temperatures indicate ignition of liquid fuel, natural gas feed is discontinued to insure sufficient air for complete combustion of kerosene. Approximately 10-15 minutes are required to raise the bed temperature to 650°C which is sufficient to achieve self-ignition of coal.

Coal, usually mixed with limestone, is then fed to the combustor from the primary injector. A steady stream of 1.7 stnd. $m^3$/min (60 scfm) of transport air is used to convey coal into the combustor. Actual rate of coal injection is determined by the pressure differential between the injector and combustor. The rate is initially set at an appropriate value based on past experience under similar operating conditions. Once ignition of coal is verified by rapidly rising temperatures, kerosene flow is stopped. At this time, the main combustion air feed line to the plenum is opened allowing most of the air to bypass the burner, and both combustion air flow rate and combustor pressure are rapidly increased to their designated operating values. Flow of water to each cooling coil is adjusted to maintain steam/water exiting temperatures of 138-150°C. Once the desired bed temperature has been reached, it is held constant by the automatic coal feed rate control system.

## Miniplant Combustor Performance

### Length of Operation and Conditions Tested--

As of June 1976, the miniplant has accumulated over 1100 hours of coal combustion time. During the shakedown phase a total operating time of 500 hours was accumulated and 37 runs conducted. Many of the initial runs were limited to 10 hours or less duration, but as the systems were improved the run durations increased. Included among the longer runs were four that were 24 hours in duration, one 50 hours in duration, and a 100 hour continuous run which concluded shakedown.

During the operations phase a total operating time of approximately 600 hours was accumulated covering 66 sets of operating conditions. Included among the longer runs were four of over 24 hours duration and a 240 hour demonstration run. During the demonstration run, coal combustion was continuous except for a total of 4 hours, during which liquid fuel was burned to maintain the bed temperature. During the last 7 days of the run, liquid fuel was used for only 15 minutes. The on line factor for coal combustion was 98.5%.

The primary objective of the operations phase of the program was to evaluate the effect of key operating parameters on the emission levels, combustion efficiency, and heat transfer rates. Table III-3 presents a brief summary of the miniplant test program, indicating the objective of each series of runs as well as the ranges of the operating variables. As indicated, the miniplant combustor proved to be very flexible and was able to run over a wide range of operating conditions.

TABLE III-3. SUMMARY OF MINIPLANT TEST PROGRAM

| Run No. | Objective | Operating Variables |
|---|---|---|
| 19.1–19.9<br>20.1–20.2<br>21<br>26 | Study the effect of temperature and the Ca/S molar feed ratio on the emission levels using Grove limestone as the sorbent | P = 930 kPa<br>Vel. = 2.0 m/s<br>Excess air = 15%<br>T = 820–954°C<br>Ca/S = 1.45–3.70<br>Expanded Bed Height = ~2–4m |
| 22 | Measure $SO_2$ level after sorbent feed stopped | P = 930 kPa<br>Vel. = 1.9 m/s<br>Excess air = 28%<br>T = 870°C<br>Ca/S = 0<br>Expanded Bed Height = 2–4m |
| 23<br>25 | Evaluate dolomites for use in the variables study | P = 930 kPa<br>Vel. = 1.9 m/s<br>Excess air = 8–12%<br>T = 870–885°C<br>Ca/S = 1.30<br>Expanded Bed Height = ~2–4m |
| 27.1–27.21 | Study the effect of temperature and the Ca/S molar feed ratio on the emission levels using Pfizer dolomite as the sorbent | P = 930 kPa<br>Vel. = 1.1–2.3 m/s<br>Excess air = 8–23%<br>T = 829–932°C<br>Ca/S = 0.0–2.5<br>Expanded Bed Height = 3–7m |
| 28.1–28.5 | Verify the accuracy of the $SO_2$ measurements by running with an inert alumina bed | P = 930 kPa<br>Vel. = 1.4–2.7 m/s<br>Excess air = 5–40%<br>T = 840–930°C<br>Ca/S = 0<br>Expanded Bed Height = 2m |

TABLE III-3 (continued). SUMMARY OF MINIPLANT TEST PROGRAM

| Run No. | Objective | Operating Variables |
|---|---|---|
| 29–30.4 | Better define the correlation between the $SO_2$ emission level and the Ca/S molar feed ratio and temperature for uncalcined limestone | P = 920 kPa<br>Vel. = 1.9–2.5 m/s<br>Excess air = 14–19%<br>T = 835–929°C<br>Ca/S = 3.7<br>Expanded Bed Height = 2–4m |
| 31<br>32.1–32.3 | Study the effect of operating pressure using limestone and dolomite | P = 520–600 kPa<br>Vel. = 1.5–2.2 m/s<br>Excess air = 13–23<br>T = 836–950°C<br>Ca/S = 0.8–2.5<br>Expanded Bed Height = 2–3m |
| 33<br>34<br>35<br>36.1–36.2<br>37<br>38.1–38.6<br>39.1–39.4 | Study the effect of secondary variables (excess air, bed depth, superficial velocity, and particle size) and evaluate the performance at turndown conditions | P = 902–932 kPa<br>Vel. = 1.3–3.0 m/s<br>Excess air = 18–112%<br>T = 674–938°C<br>Ca/S = 0.8–2.5<br>Expanded Bed Height = 2–4m |

Control of Operating Conditions--

Good control of each of the operating variables was demonstrated for sustained periods. Presented below are the averages and standard deviations calculated for the combustor operating conditions over a 1 hour interval during steady state conditions for a typical combustor run:

| Variable | Average | Standard Deviation |
|---|---|---|
| Bed temperature (°C) | 901 | 3 |
| Coal feed rate (kg/hr) | 119 | 6 |
| Combustion pressure (kPa) | 920 | 1 |
| Gas superficial velocity (m/s) | 3.1 | 0.02 |
| Bed height (m) | 6.7 | 1.3 |

Bed temperatures were well controlled using the control system described earlier. Very good temperature profiles were established with temperature variations across the bed generally being approximately 30°C. The temperatures of the flue gas exiting the cyclones were typically close to the temperatures at the top of the expanded bed.

The combustor pressure was well controlled using a fixed converging nozzle with supplementary air addition. No pressure control problems were noted at pressures between 520 and 930 kPa. Steady gas velocities were maintained by achieving good control of the air flow rate, pressure, and bed temperature.

The combustor bed level was well controlled by continuously removing solids from the combustor through a pulse pot solids reject system as described on page . The pulsing rate was adjusted to maintain a nearly constant bed level as indicated by a constant pressure drop across the bed.

Cooling Coils--

The design of the miniplant cooling coils has undergone a number of modifications in an attempt to overcome problems of erosion and fatigue and to promote better solids mixing. A detailed description of the coils used through Run 19.3 has already been presented in an earlier report (2).

Since then, new coils were constructed from 1.3 cm schedule 40 316 stainless steel pipe for protection against fatigue and, in the event of bed agglomeration, against deformation. Support rods connecting the inlet and outlet piping to the adjacent coils were used to hold the coils rigid. Pipe bends having a 35 mm radius and 170 mm long tangent were butt welded together in the construction of the coils. The avoidance of socket welded U bends as used in prior coils allowed fewer welds. Also, the sockets of the U bends were susceptible to damage from erosion and/or corrosion. The surface area of each coil was 0.55 m², approximately the same area as used in the previous coils.

These changes significantly lengthened the lifetime of the coils, but the coils were still susceptible to damage. These coils failed after a total operating time of 140 hours. The damage was localized at several of upper U bends located towards the interior of the column. These U bends had holes and eroded surfaces on the under side of the U bends. The photograph in Figure III-8 is a typical example. It is believed that the erosion occurred because jets of gas bypassed the bed by forming high velocity annular gas streams along the length of the vertical tubes. Bed solids accelerated by the gas stream impinged on the underside of the upper U bends. Velocities of 15-30 m/sec or more must have been attained to explain the erosion seen.

Prior to the 240 hour demonstration run, the coils were baffled with bars on the underside of the more susceptible U bends. After the run it was discovered that the cooling coils had again sustained damage due to erosion. The most extensive damage was exhibited by coil 1B. Coil 2A also showed signs of erosion, but not as severe as coil 1B. Damage was not detected on coil 1A and only a few eroded surfaces were found on coil 2B. Also, many of the welds connecting the vertical tubes located near the center of the column were eroded to a smooth surface. The erosion was again attributed to gas bypassing.

In the next effort to protect the coils by diverting the gas jets, baffles were installed on the coils by welding 2.3 X 4.2 cm rings on each tube, one each at the upper and lower U bends and two at intermediate positions. Half rings were primarily used, staggered at 90° intervals about the vertical axis. The rings on adjacent tubes were staggered to maximize the cross sectional area vailable to the upward moving solids and gases at any position along the tube bank. The rings proved to be very effective and no signs of erosion were noted after more than 200 hours of operation. Figure III-9 shows a photograph of a typical coil after 15 hours of use. It was subsequently found that it was only necessary to use half rings at the upper U bend to protect the coils. Coils built recently have used only the upper half rings.

Temperature Distribution--

As discussed in an earlier report, a dramatic improvement in the bed temperature profile was observed when vertically oriented cooling coils were used (2). Also noted was a substantial increase in the freeboard temperature when beds depths were increased to immerse all coils and prevent cooling of the flue gas above the expanded beds. Flue gas temperatures comparable to the combustion zone temperatures were thus achieved.

When baffles were added to the cooling coils to prevent erosion, the possibility that the baffles would affect the quality of solids mixing and the heat removal capacity of the coils was considered. Typical temperature profiles for baffled and unbaffled coils are shown in Figure III-10. The temperature drop across the bed was slightly greater with the baffled coils (35°C vs 27°C), but the difference is of negligible significance. The average heat transfer coefficient for the baffled coils compared very closely with the average coefficient for the unbaffled coils. The baffles which act as fins added about 15% to the surface area of the coils. It is likely, however, that the stagnant areas created by the baffles reduce the bed to tube heat transfer coefficient. The competing effects may cancel, resulting in little or no change in the heat removal capacity.

Particulate Sampling System--

The isokinetic particulate sampling system presently being used was shown schematically in an earlier section of the report. To assure reliable, and accurate particulate emission measurements, the sampling system was

FIGURE III-8

EROSION DAMAGE AT THE UPPER U BEND OF COIL 1B

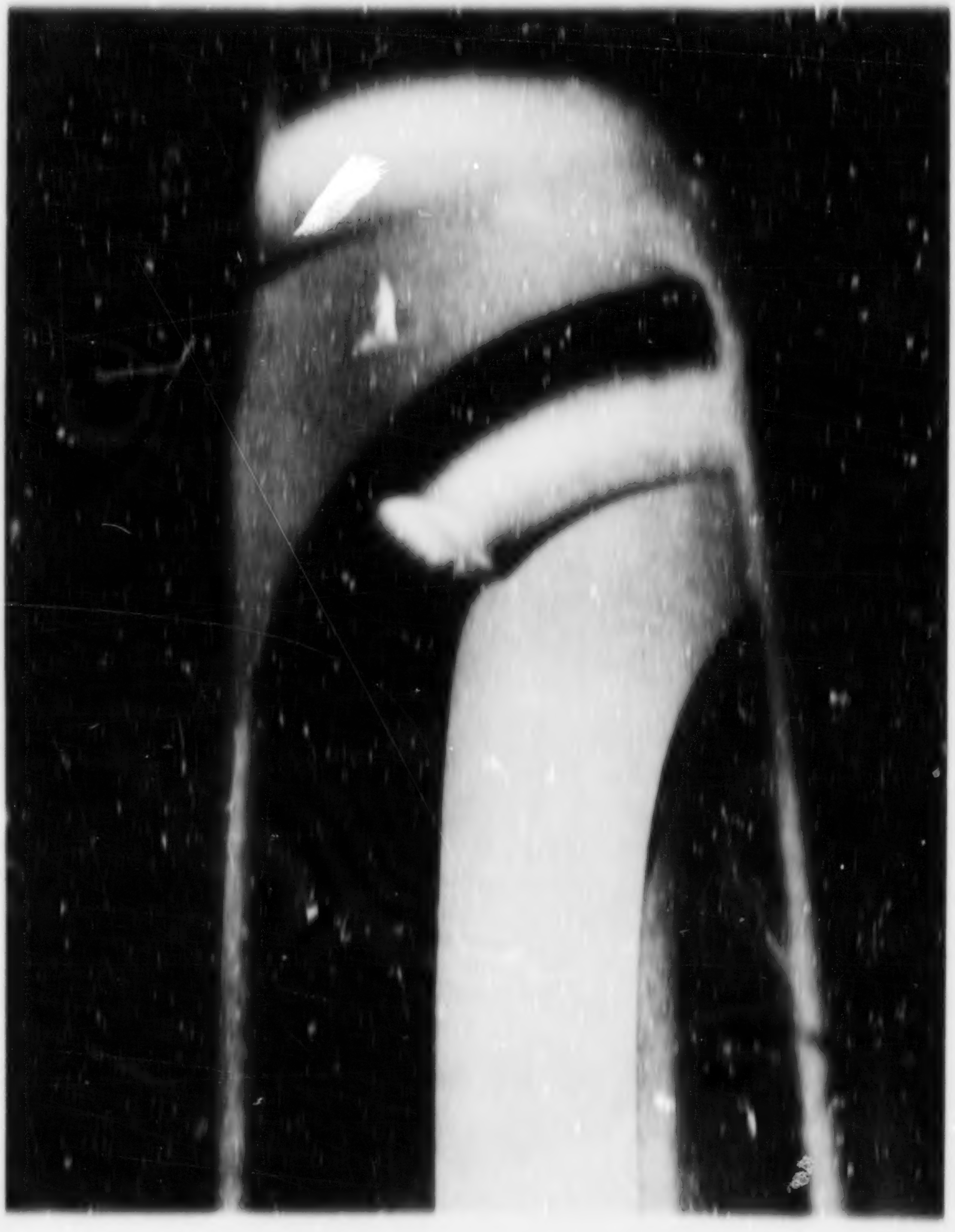


FIGURE III-9

BAFFLED COIL AFTER 15 HRS OF OPERATION

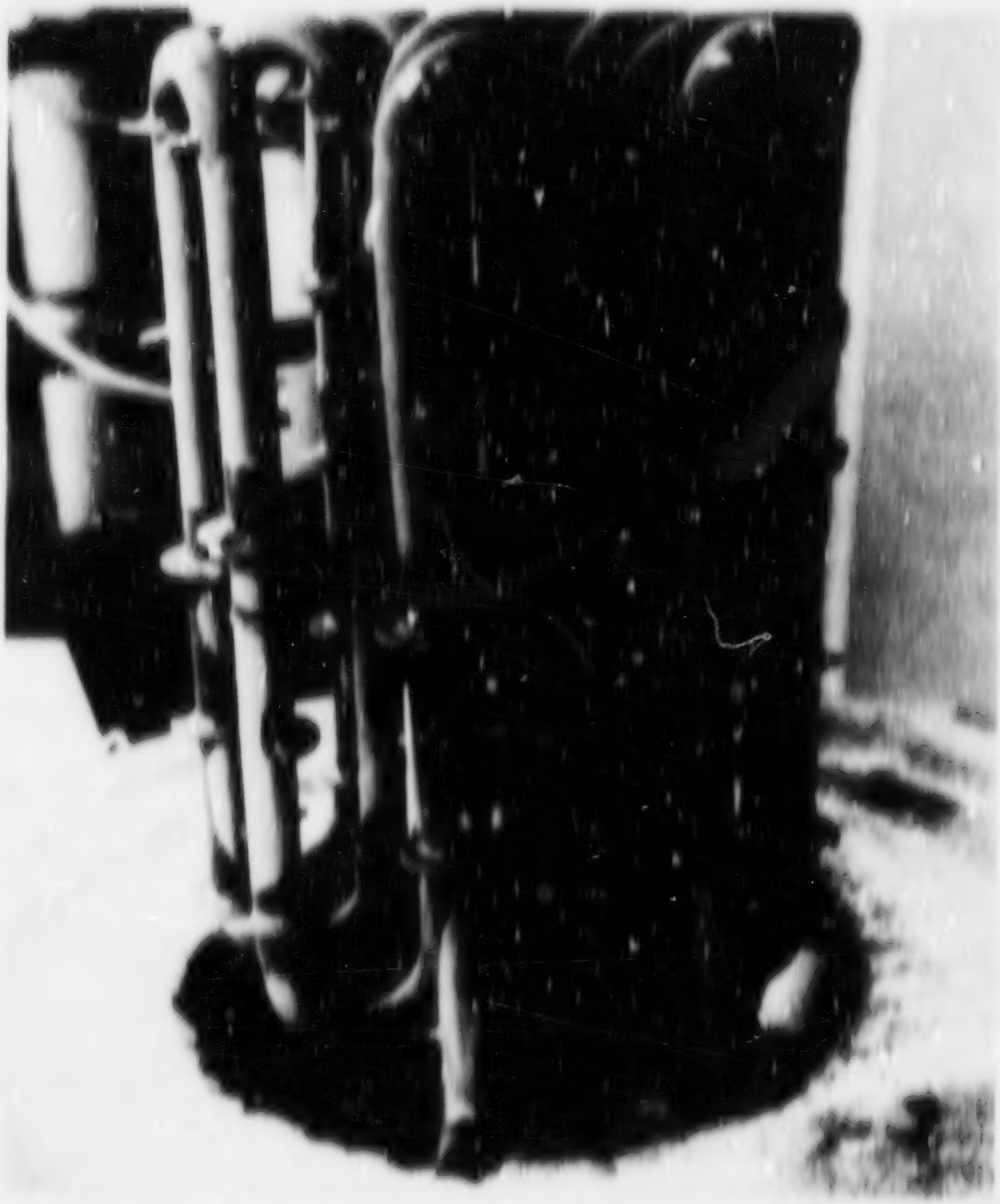
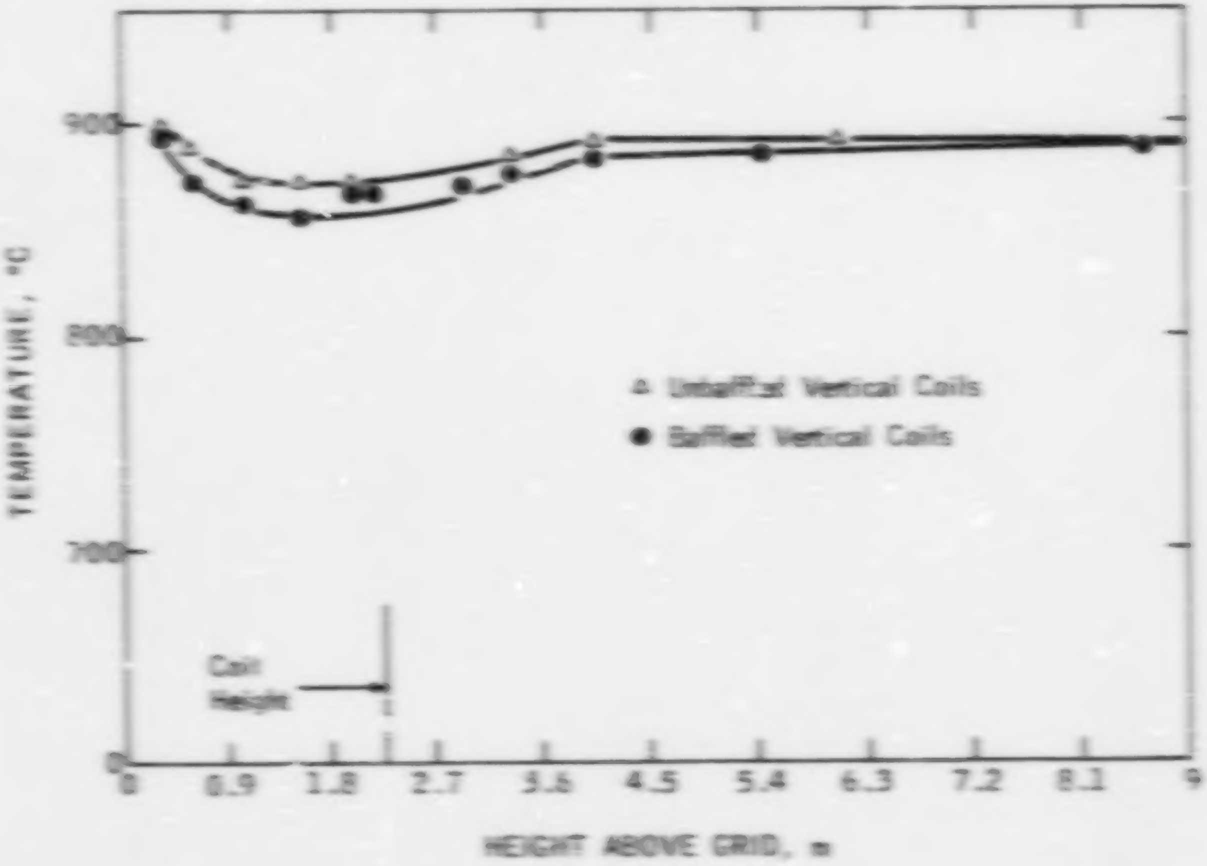


FIGURE III-10

MINIPLANT COMBUSTOR TEMPERATURE PROFILE



continually modified and improved. Poor performance of earlier systems were caused primarily by:

- plugging problems
- temperature control problems
- moisture condensation problems

A comparison of the performance of earlier sampling systems with the present one shows a substantial reduction in the deviation from isokinetic sampling using the present system. Other modification are planned to further reduce deviations from isokinetic sampling and to simplify operation.

The present sampling system also allows the use of an impingement type device to measure the flyash particle size distribution. The major advantage in using this type of device is that it gives a direct measurement of the size distribution as compared to systems which rely on collection-redispersion techniques.

## EXPERIMENTAL RESULTS

The primary objective of the experimental program was to measure and compare the performance of a limestone and dolomite sorbent for $SO_2$ control over a range of operating conditions. Variables included the sorbent feed rate, pressure, bed temperature, superficial velocity, bed depth, excess air level and sorbent feed particle size. The bulk of the experimental work focused on the effect of the primary variables, the sorbent feed rate and bed temperature. In addition to $SO_2$ control, a number of other measures of process performance were determined as a function of operating conditions. This included $NO_x$, CO, $SO_3$ and particulate emissions, attrition and elutriation of the sorbents, carbon combustion efficiency and heat transfer coefficients between the fluidized bed and the cooling coils.

The effect of the primary variables on the performance of Grove No. 1359 limestone was investigated in a series of 15 runs encompassing 26 test conditions. The Ca/S molar feed rate was varied from 1.5 to 3.7 and the temperature was varied from 840 to 950°C. The runs lasted from 6 to 16 hrs., except for one 36 hour run. The effect of the primary variables on the performance of Pfizer No. 1337 dolomite was investigated during Run 27, which was a 240 hour continuous run. Twenty-one test conditions were completed during Run 27. The Ca/S molar feed ratio was varied from 0.6 to 2.2, and the temperature was varied from 840 to 950°C.

Runs examining secondary variable effects were also run. Runs 31 and 32.1-32.3 were made at a reduced pressure of 500 to 600 kPa, compared to 900 to 940 kPa used in all other runs. Both limestone and dolomite were used. The effect of sorbent particle size on $SO_2$ emissions was measured in runs 38.1, 38.2, 39.3 and 39.4. The standard 8 to 25 mesh sorbents were screened into two size fractions, 8 to 14 mesh and 14 to 25 mesh. This gave a 2/1 ratio of mass average particle sizes in the two fractions. The effect on $SO_2$ emissions of varying the gas residence time by changing the gas velocity and bed height were examined in runs 33 to 37. Velocities up to the design value of 3 m/sec were used. Excess air levels as great as 100% were attained in these tests because of a reduction in the heat transfer surface area. Pfizer dolomite was the only sorbent used in runs 33 to 37.

The operability of the unit at "turndown" temperatures of 690 and 760°C and the effectiveness of limestone and dolomite as sorbents at these temperatures was investigated in runs 38.1 to 38.6, 39.3 and 39.4.

Run 28 was made with an inert bed of alumina to establish the accuracy of the monitored $SO_2$ emission levels by comparison with that value calculated from the sulfur content of the coal and the excess air level.

The experimental program is summarized in Table III-4. Comprehensive information on the individual runs is available in Appendix B.

## Calculation of Ca/S Ratio by Sulfur and Calcium Balance

A sulfur and calcium balance around a combustor gives the expression:

$$\text{Ca/S (Mole/Mole)} = \frac{\text{Sulfur Retention (\%)}}{\text{Calcium Utilization (\%)}}$$

This is an exact expression when all input and output streams are included.

At steady state (feed rates constant, bed height and composition constant, output rates and compositions constants, etc.) the retention and utilization will be a function of the Ca/S ratio (and other variables such as residence time and temperature). The time required to displace all the solids in the bed is long, possibly on the order of 20 to 40 hours. This is based on the fact that the solids residence time is usually in the range of five to ten hours and complete replacement of the solids requires four times the residence time. Therefore, if operating conditions in the combustor are changed significantly, a very long line-out period would be required to assure that the solids composition in the combustor has reached the new steady state value. In practice, the line-out time is usually on the order of two to four hours and is based on the time required for the $SO_2$ content in the flue gas to reach a new relatively constant value. Therefore, the solid composition may not always have achieved its new steady state level when the data gathering portion of the new run begins.

Since it was impractical to wait until the solids has reached steady state in each run, it was felt that a more significant correlation could be obtained by calculating a Ca/S ratio using the retention and bed utilization that existed at the time the data were being collected.

In practice, the use of the calculated Ca/S ratio has given somewhat better correlations than use of the "set" value of the Ca/S ratio. The "set" Ca/S ratio is that based on the settings of the limestone and coal feed screws. This is especially true in cases where the set Ca/S ratio was changed radically between runs. In other cases (e.g., runs 30.1, 30.2 and 30.4) obvious mechanical problems prevented an accurate value of Ca/S (set) from being obtained. Generally it has been found that use of the calculated Ca/S ratio rather than the set value shows a somewhat higher (20%) sorbent requirement.

TABLE III-4. MINIPLANT COMBUSTION PROCESS VARIABLE STUDIES

| Test Series | Run No. | Pressure (kPa) | Temp. (°C) | Ca/S (Mole/Mole) | Super. Velocity (m/s) | Settled Bed Depth (m) | Excess Air (%) | Feed Sorbent Part. Size (Mesh) |
|---|---|---|---|---|---|---|---|---|
| Primary Variables | | | | | | | | |
| Grove Limestone | 19.1-22<br>26-29<br>30.1-32.4 | 930 | 820-940 | 1.45-3.7 | 1.7-2.3 | .7-2.3 | [illegible]-28 | 8 X 25 |
| Pfizer Dolomite | 27.1-27.22 | 930 | 930-930 | .35-2.5 | 1.1-2.2 | 2.2-3.9 | 8-23 | 8 X 25 |
| Secondary Variables | | | | | | | | |
| Grove Limestone | 31, 37.1,<br>39.3, 39.4 | 520, 600<br>930 | 840-950 | 2.5 | 1.6-2.2 | 1.3-1.9 | 13-32 | 8 X 25<br>8 X 14, 14 X 25 |
| Pfizer Dolomite | 32.1-38.2 | 600, 930 | 840-950 | .75-2.5 | 1.5-3 | 0.8-2.3 | 16-96 | 8 X 25, 8 X 14<br>14 X 25 |
| Turn Down, Heat Transfer | | | | | | | | |
| Grove Limestone | 39.1, 39.2 | 902 | 670-760 | 2.5 | 1.4-1.5 | 1.2 | 60-63 | 8 X 25 |
| Pfizer Dolomite | 38.3-38.6 | 930 | 690-760 | 0.75-1.5 | 1.3-1.85 | 1.1-2.6 | 80-112 | 8 X 25 |
| Inert Bed | | | | | | | | |
| (Alumina) | 28.1-28.5 | 930 | 840-930 | 0 | 1.4-2.65 | 1 | 5-40 | 8 X 25 |

obtained. Generally it has been found that use of the calculated Ca/S ratio rather than the set value shows a somewhat higher (20%) sorbent requirement.

As more data become available from the combustor, attempts will continue to be made to find the "best" means of correlating the retention with the measured parameters even when the bed composition has not reached a steady state. In this report, the calculated Ca/S ratio is used unless otherwise noted. However, the set values have also been included in the run summary tables.

## Control of $SO_2$ Emissions with Pfizer No. 1337 Dolomite

### Effect of Ca/S Molar Ratio--

Pfizer dolomite is a very active sorbent. During Run 27, the $SO_2$ emission level was reduced to below 100 ppm at a Ca/S ratio of only 2.0, as shown in Figure III-11. If the data are replotted as sulfur retention vs. Ca/S ratio, as in Figure III-12, it is seen that the retention levels exceeded 95% at a Ca/S ratio of 2.0. The EPA $SO_2$ emission standard of 1.2 lb $SO_2$/M BTU was met for the 2% sulfur Champion coal with a Ca/S mole ratio of only 0.85, corresponding to a 60% retention level. An examination of the retention data shows that high calcium utilizations were achieved. At Ca/S ratios less than 1.0, the utilization levels are in the range of 50% to 90%. In this region the retention level decreases linearly with the Ca/S mole ratio. The lowest utilization level was ~45% at a Ca/S ratio of 2.0

### Effect of Dolomite Utilization on the Rate of Sulfation--

The rate at which calcium reacts with $SO_2$ under fluidized bed combustion conditions may be described by a reaction rate model which is first order in $SO_2$ concentration (4,5).

The sulfation reaction rate constant can be calculated from experimental sulfur retention levels using the equation:

$$\text{First order rate constant, k, } (s^{-1}) = -\frac{1}{t_{gas}} \ln \left(\frac{100-\text{Retention}}{100}\right)$$

where $t_{gas}$, the gas phase residence time is given by

$$t_{gas} = \frac{\text{Expanded bed height (m)}}{\text{Superficial gas velocity (m/s)}}$$

The rate constant decreases with the sorbent utilization because the decreased porosity limits access to the active sites. This dependence is shown in Figure III-13. Data were included from units other than the mini-plant to obtain a greater number of data points over a wider range of utilizations and to judge the consistency of the data which have been generated to date. Irrespective of the differences shown in Table III-5 with regard to the reactor geometry, temperature, pressure, particle size, and dolomite and coal type, a good correlation was obtained. The fraction of the explained variance was 88% and the F ratio was 154. The correlation was refined by rejecting 4 of the 47 points which were beyond the 2σ limit on the first regression. The data were also examined for the effect of temperature and pressure but neither was statistically significant. Two primary conclusions are that the agreement between data from different units is good and that the effect of

FIGURE III-11

MINIPLANT $SO_2$ EMISSIONS FOR DOLOMITE NO. 1337

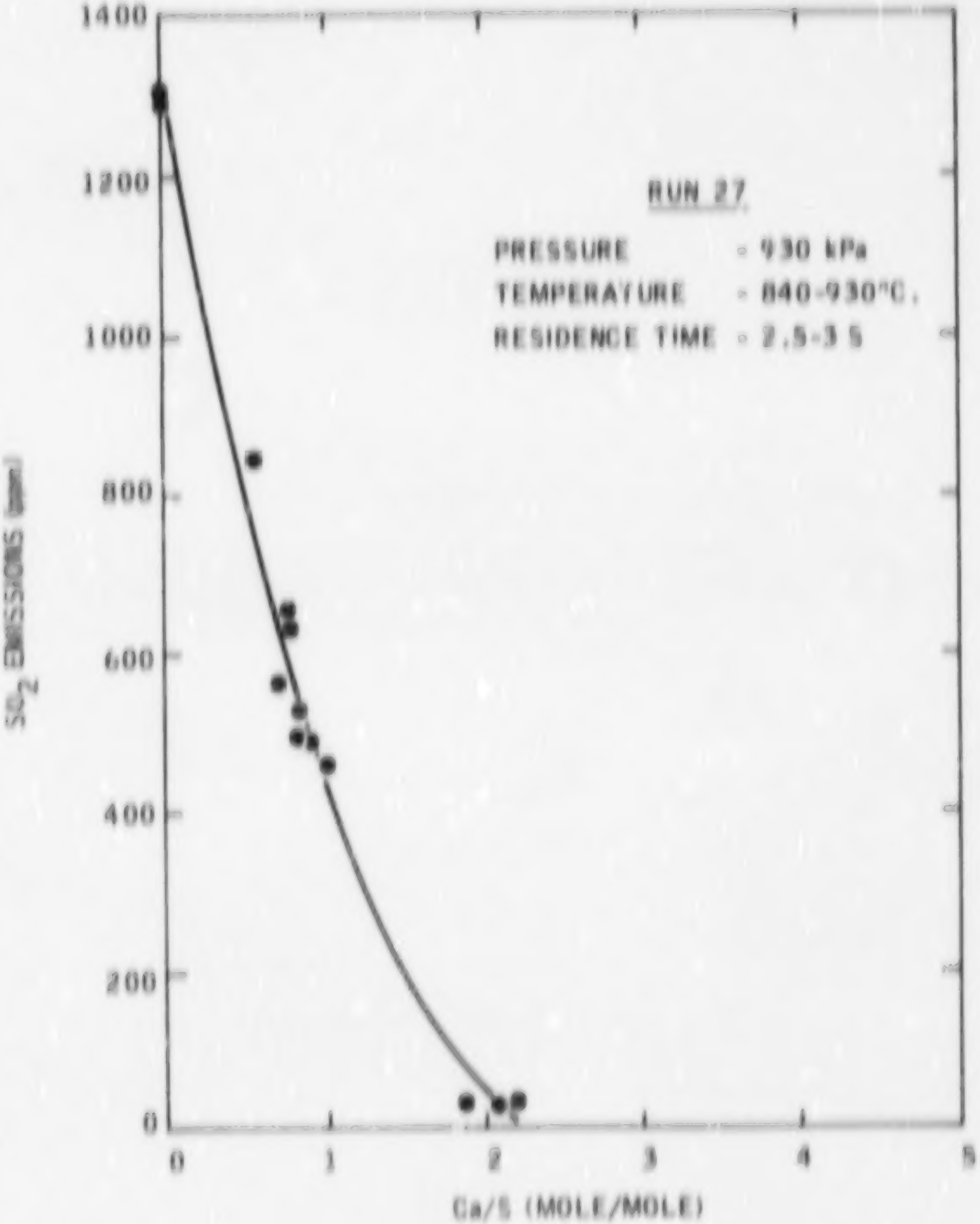


FIGURE III-12

MINIPLANT $SO_2$ RETENTION VS. Ca/S RATIO FOR DOLOMITE NO. 1337

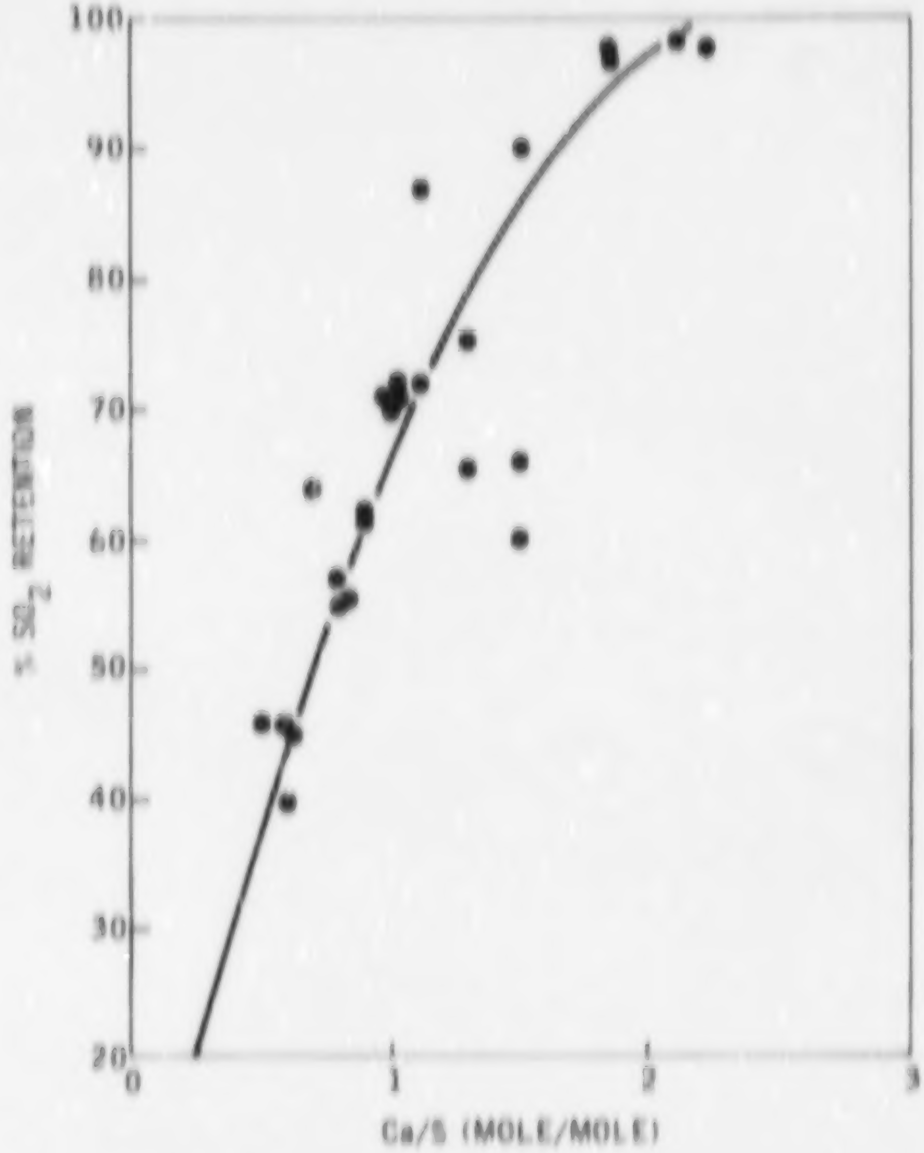


TABLE III-5. COMPARISON OF FBC PRESSURIZED UNITS

| Laboratory | Unit Size (m) | Tests | Press. kPa | Temp. °C | Gas Residence Time (sec) | Ca/S (Mole/Mole) | Sorbent | Coal | Bed Mass Avg. Part. Size (μm) |
|---|---|---|---|---|---|---|---|---|---|
| Argonne[6] | 0.15 dia. | VA01/VA09 | 810 | 785–900 | 0.6–1.4 | 1–3.2 | T DOL | ARK | 800 |
| | | EA1/EA9 | 810 | 900 | 0.7 | 1.1–2.9 | T DOL | ARK | 590 |
| BCURA[7] | 0.6 x 0.9 | 1, 2, 3.1,<br>3.2 | 600 | 890–950 | 2 | 1.6–2.2 | P DOL | PITTS & ILL. | 800 |
| BCURA[8] | 1.2 x 0.6 | 1.1/1.5 | 350 | 800 | 1.9 | 0.7–0.9 | UK DOL | WELBECK | 740 |
| | | 2.1/2.4 | 350 | 800 | 1.9 | 1.6–1.95 | P DOL | PITTS | 1200–1500 |
| | | 3.1 | 510 | 800 | 1.9 | 1.5–2.0 | P DOL | PITTS | 900 |
| | | 3.2 | | | | | | | |
| | | 4.1, 4.2 | | | | | | | |
| EXXON | 0.3 dia.<br>Miniplant | 27.1/27.21<br>32.2, 32.3<br>35, 36.1, 36.2<br>37 | 930 | 840–950 | 1–3 | 0.5–2.5 | P DOL | CHAMP | 800–1200 |

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T DOL - Tymochtee dolomite
P DOL - Pfizer No. 1337 dolomite
UK DOL - United Kingdom dolomite
ARK - Arkwright
PITTS - Pittsburgh
ILL - Illinois
CHAMP - Champion

temperature and pressure is minor. Utilizations as great as 70-80% were commonly obtained in the miniplant when deep beds were used in conjunction with low Ca/S ratios. The utilizations in the Argonne unit were lower, mostly 35-50%, primarily because of shallow beds and low residence times. The NCURA data overlaps the data from the other two units. However, most of the utilizations range between 45-60%.

Effects of Other Variables--

Temperature-- Statistical analysis of emission data from Run 27 where the temperature ranged from 840-950°C did not reveal a temperature effect on activity, nor did statistical analysis of the first order rate constant vs. utilization data given in Figure III-13. Emission data from "turndown" runs 38.3 to 38.6 did, however, show that activity was slightly lower at the reduced temperature levels of 690°C and 760°C. The turndown data are compared to data from Run 27 in Figure III-14.

Parameters which may affect activity and have a temperature dependence are the intrinsic rate constant, diffusivity and pellet porosity. The porosity depends on the degree of calcination. Depending on the operating conditions, dolomite undergoes either half calcination or full calcination when fed to the combustor. At a pressure of 930 kPa, the temperature required for full calcination is ~900°C. Many tests were made under conditions which were marginally unfavorable for full calcination. In the case of the turndown tests, they were very unfavorable. The high activity at turndown temperatures shows that half calcined dolomite is sufficiently porous to have good activity and that the reaction between calcium carbonate and sulfur dioxide is rapid. The small temperature dependence over a fairly broad temperature range is characteristic of diffusion controlled reactions.

Gas Residence Time--The effect of gas residence time on sulfur retention was investigated in Runs 35 to 37. The gas velocity and bed height were varied to obtain residence times between 1 and 3 s. The emissions data are plotted in Figure III-15 together with the retention profile obtained from Run 27 where the gas residence time was 2.5-3 s. As expected, the retention levels of the low residence time data generally falls below the profile from Run 27.

A prediction of the residence time effect can be made using the following approach. A sulfur balance gives:

$$Ca/S = \frac{SO_2 \text{ Retention } (\%)}{\text{Calcium Utilization } (\%)}$$

The $SO_2$ retention level, R, is derived from first order kinetics to be:

$$R = 100\ (1-e^{-kt_{gas}})$$

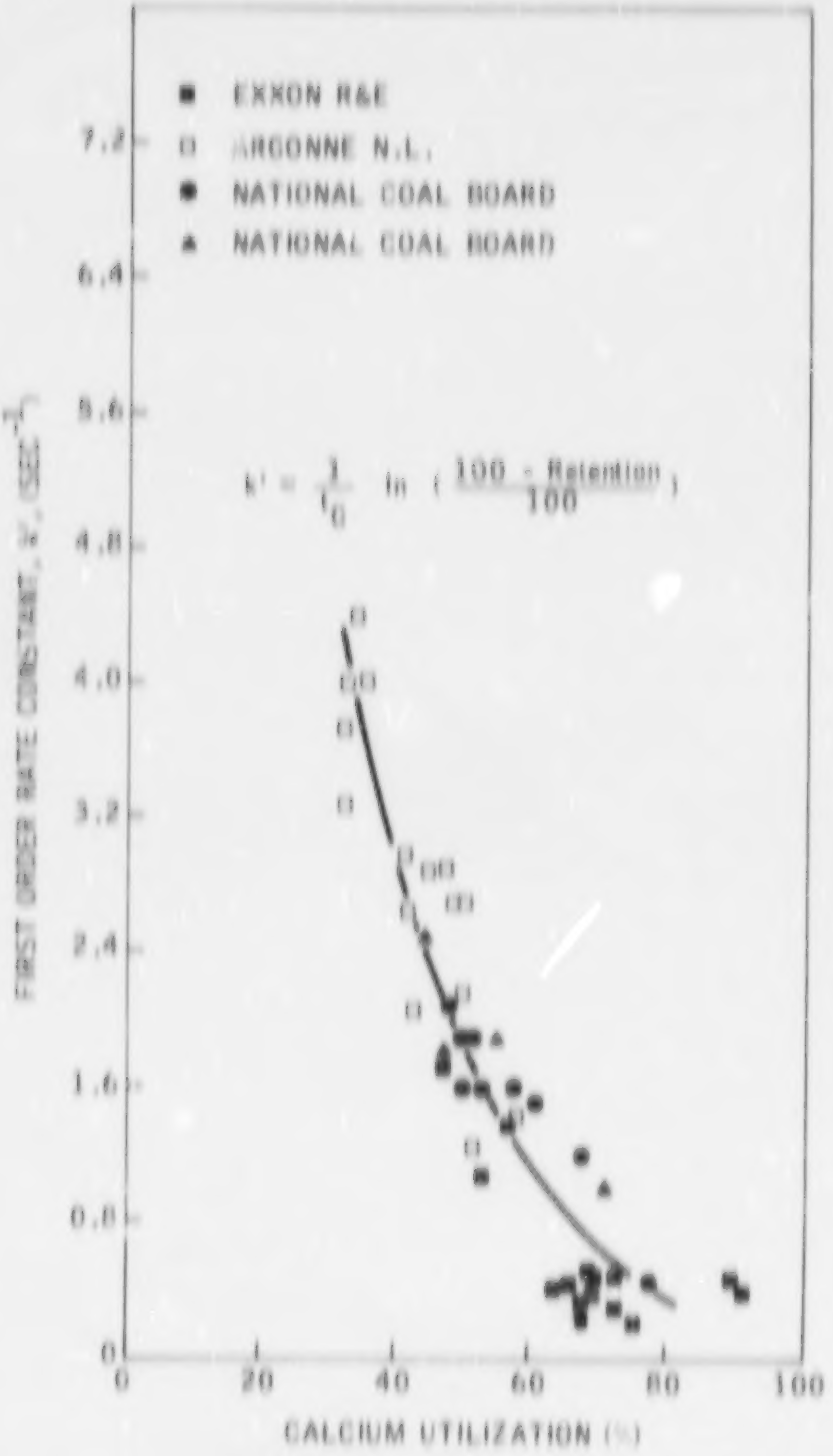
and the rate constant, k, as a function of utilization can be obtained directly from Figure III-13 or can be calculated using the empirical equation:

$$k = 10.11 - 0.234\ (\text{utilization}) + 0.0014\ (\text{utilization})^2$$

These three equations with five variables have two degrees of freedom. Therefore, if two of the variables are set, such as residence time and Ca/S ratio,

## FIGURE III-13

## ACTIVITY VS. CALCIUM UTILIZATION FOR DOLOMITE



## FIGURE III-14

## EFFECT OF TEMPERATURE AND SORBENT PARTICLE SIZE ON $SO_2$ RETENTION - DOLOMITE No. 1337

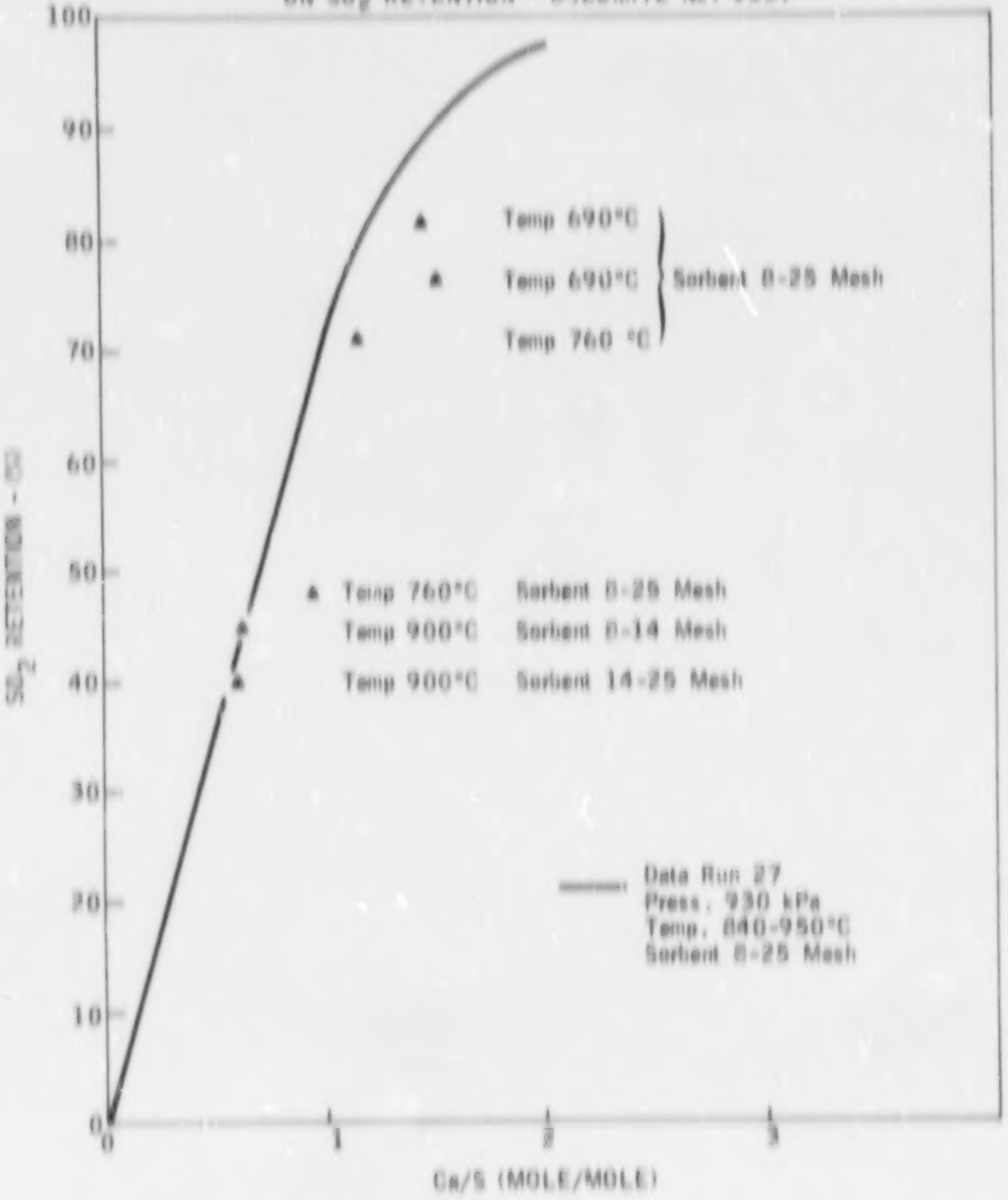
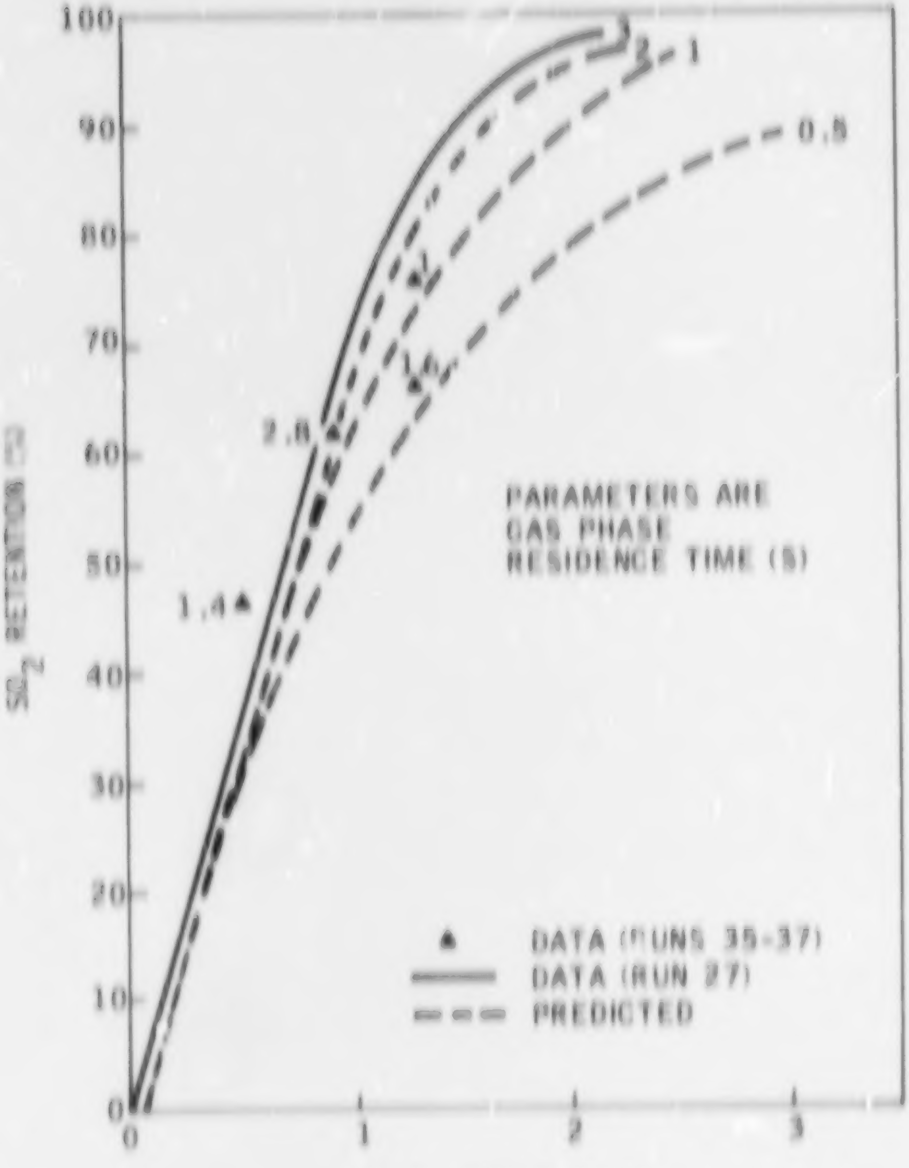


FIGURE III-15

EFFECT OF GAS RESIDENCE TIME ON $SO_2$ RETENTION
DOLOMITE NO. 1337



the system is defined and the other three variables, rate constant, utilization and retention can be calculated. The calculation is a trial and error procedure. Predicted retention profiles generated for residence times of 0.5, 1 and 2 s are included in Figure III-15. The profiles do not differ much in the 1-3 s range. This is especially true at Ca/S ratios below 1.0 where the profiles converge. The effect does become increasingly important, however, when the residence time is below 1.0 s and must be considered when very shallow beds are employed. A comparison of the emissions data to the predicted retention levels is given in Table III-6.

TABLE III-6.

| Run No. | Gas Residence Time (s) | Measured Retention (%) | Predicted Retention (%) |
|---|---|---|---|
| 32.2 | 0.8 | 60 | 75 |
| 32.3 | 0.8 | 66 | 75 |
| 35 | 2.8 | 62 | 64 |
| 36.1 | 1.6 | 66 | 75 |
| 36.2 | 1.0 | 76 | 73 |
| 37 | 1.4 | 46 | 35 |

The measured retention levels from runs 35 to 37, made at 9 atm total pressure are in good agreement with the predicted retention levels. The data from Runs 32.2 and 32.3 could be low because of the reduced pressure of 600 kPa.

The dependence on gas residence time of the Ca/S ratio required to meet the EPA $SO_2$ emission standard of 1.2 lb $SO_2$/M BTU is shown in Figure III-16. These curves were calculated using the above equations. The effect is not large, but is evident. As an example, for a 2% sulfur coal, an increase in gas residence time from 1 s to 3 s reduces the Ca/S requirement from ~0.95 to ~0.75.

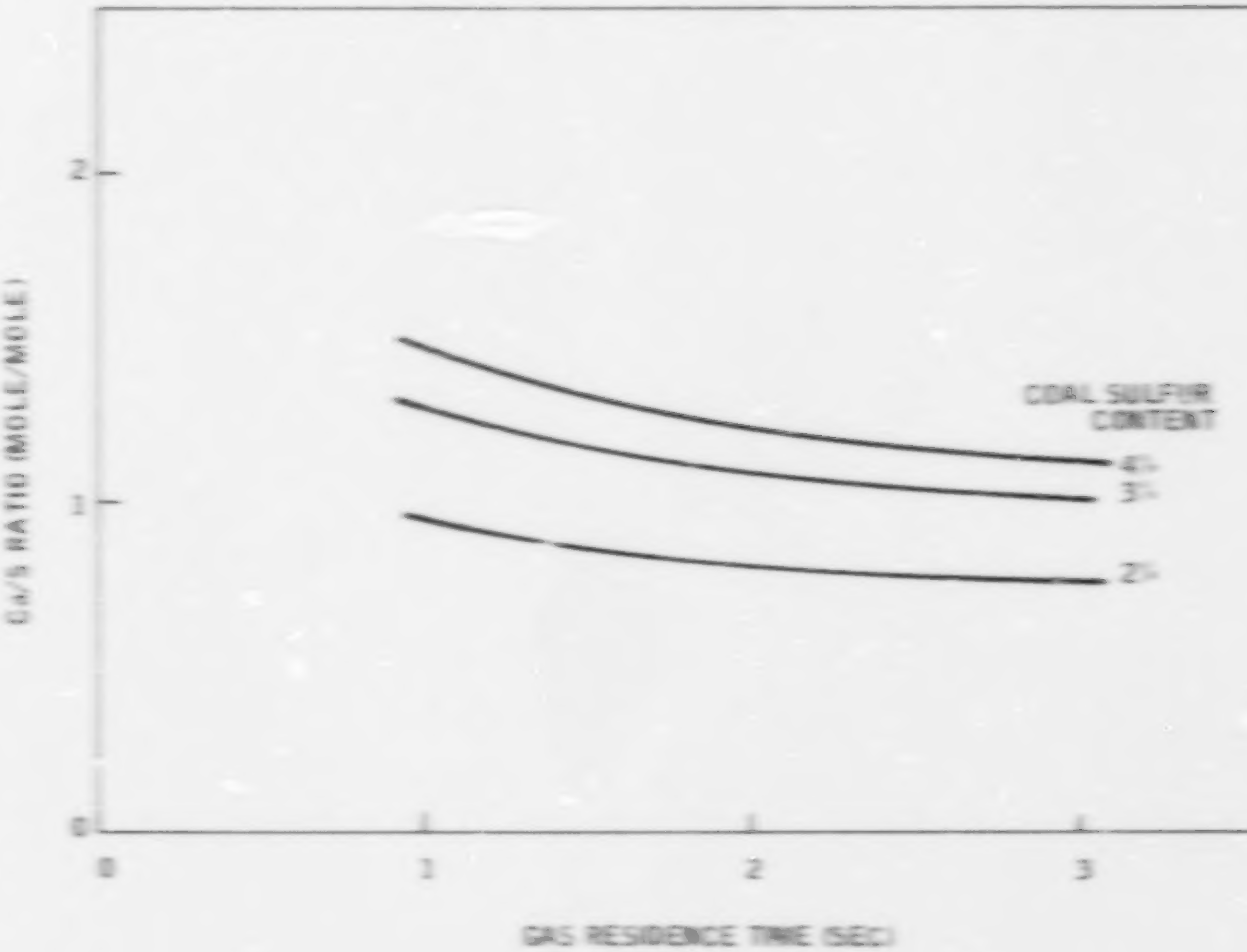
Particle Size Effect-- The particle size effect was examined in Runs 38.1 and 38.2. A dolomite feed stream with a size range of 8-14 mesh was used in one test while the other test used a 14-25 mesh feed. The mass average size ratio of the two feed streams was ~2. The emission level in the two tests did not differ significantly as is shown in Figure III-14. The final size distribution of the bed material in Runs 38.1 and 38.2 could not be determined. An operating problem with the bed removal system did not allow a sample to be taken. The mass average size of the beds could very well have been more similar than that of the feed streams. Since the dolomite attrits more readily.

Pressure and Excess Air--No pressure or excess air dependencies were identified when the data in Figure III-13 were examined by regression analysis. The pressure ranged from 350 kPa in the BCURA 1.2 X 0.6 m unit to 930 kPa in the miniplant. Excess air effects were not evident in the data from Run 27 either, when those data were examined alone.

The $SO_2$ retentions were lower than expected in Runs 32.2 and 32.3 made at a reduced pressure, 600 kPa. However, insufficient data are available at low pressure operations to determine if a pressure dependence exists.

FIGURE III-26

EFFECT OF GAS RESIDENCE TIME ON Ca/S RATIO REQUIRED TO MEET EPA $SO_2$ EMISSION STANDARD WITH DOLOMITE



## Control of $SO_2$ Emissions with Grove No. 1359 Limestone

### Effect of Ca/S Ratio and Temperature--

$SO_2$ emissions for runs using Grove limestone are shown in Figure III-17 as a function of the Ca/S molar feed ratio. Data were obtained at a pressure of 930 kPa, at Ca/S molar feed ratios of 1.1 to 4 and over a temperature range of 825 to 930°C. The emissions were significantly higher at temperatures less than 900°C because of the stone's inability to calcine extensively. Calcination greatly increases the stone's porosity making the active sites more accessible to the $SO_2$ reactant. In the following discussion and figures, calcining conditions refer to combustion temperatures above 900°C while carbontaing conditions refer to temperatures below 900°C. The data in Figure III-17 are replotted in Figure III-18 as $SO_2$ retention vs. Ca/S molar feed ratio. A 60% retention level was required to meet the EPA $SO_2$ emission standards of 1.2 lb $SO_2$/M BTU for the 2% sulfur Champion coal. The standard was met with a Ca/S ratio of approximately 1.4 under calcining conditions and approximately 2.3 under carbonating conditions.

### Calcination Effects--

The Grove limestone variable study runs showed that the bed would undergo full calcination when the temperature was raised from 880°C to 930°C. Correlations of the dissociation pressure of $CaCO_3$ as a function of temperature predict that calcination would have begun at a temperature of 916°C (see Figure III-19). The composition of the bed during the interval of steady state operation is given in Table III-7 for many of the runs. At the lower temperatures, the bed was largely composed of $CaCO_3$ and $CaSO_4$. Limited calcination did occur near the fluidizing grid in a zone where the $CO_2$ concentrations were low. The extent of calcination of a particle with each pass through the calcining zone depends upon the particle's residence time in that zone, upon the $CO_2$ concentration profile and on the thickness of the sulfation shell. A working model describing the progress of the calcination and sulfation reactions is shown in Figure III-20. Calcination occurs at the boundary between the reaction shell and carbonate core. When a particle has been recently introduced into the combustor, $CO_2$ within the particle can readily diffuse from the particle and calcination proceeds rapidly when the particle is in the calcining zone. Once the particle has been sulfated, however, the $CO_2$ diffusion and calcination rates are much slower. The increased porosity at the point of calcination makes the CaO accessible to $SO_2$, however, $CO_2$ also competes for these sites once the particle reenters the carbonating zone. The relative rates of the sulfation and carbonation reactions in part, determine how rapidly limestone sulfates. The particles reside mostly in the carbonating zone. Therefore, much of the CaO liberated by calcination is reacted before the particles reenter the calcining zone. The reaction zone in the more highly sulfated particles is likely small and the rate of calcination might be the rate determining step. The operating conditions can affect the solids recirculation rate between calcining and carbonating zones, the extent of the calcining and carbonating zones, and the $CO_2$ concentration profile. All of these factors affect the extent of calcination. As an example, a higher gas velocity increases the recirculation rates, causing a particle to see the calcining zone more frequently.

The utilizations in the shell of sulfation must range from 60-90% to account for the overall utilizations of 20-30%. A greater porosity near the particle's exterior and a more favorable pore structure generated by a slow vs. fast calcination step may make the high utilizations possible.

FIGURE III-17

$SO_2$ EMISSION VS. Ca/S RATIO FOR LIMESTONE NO. 1359

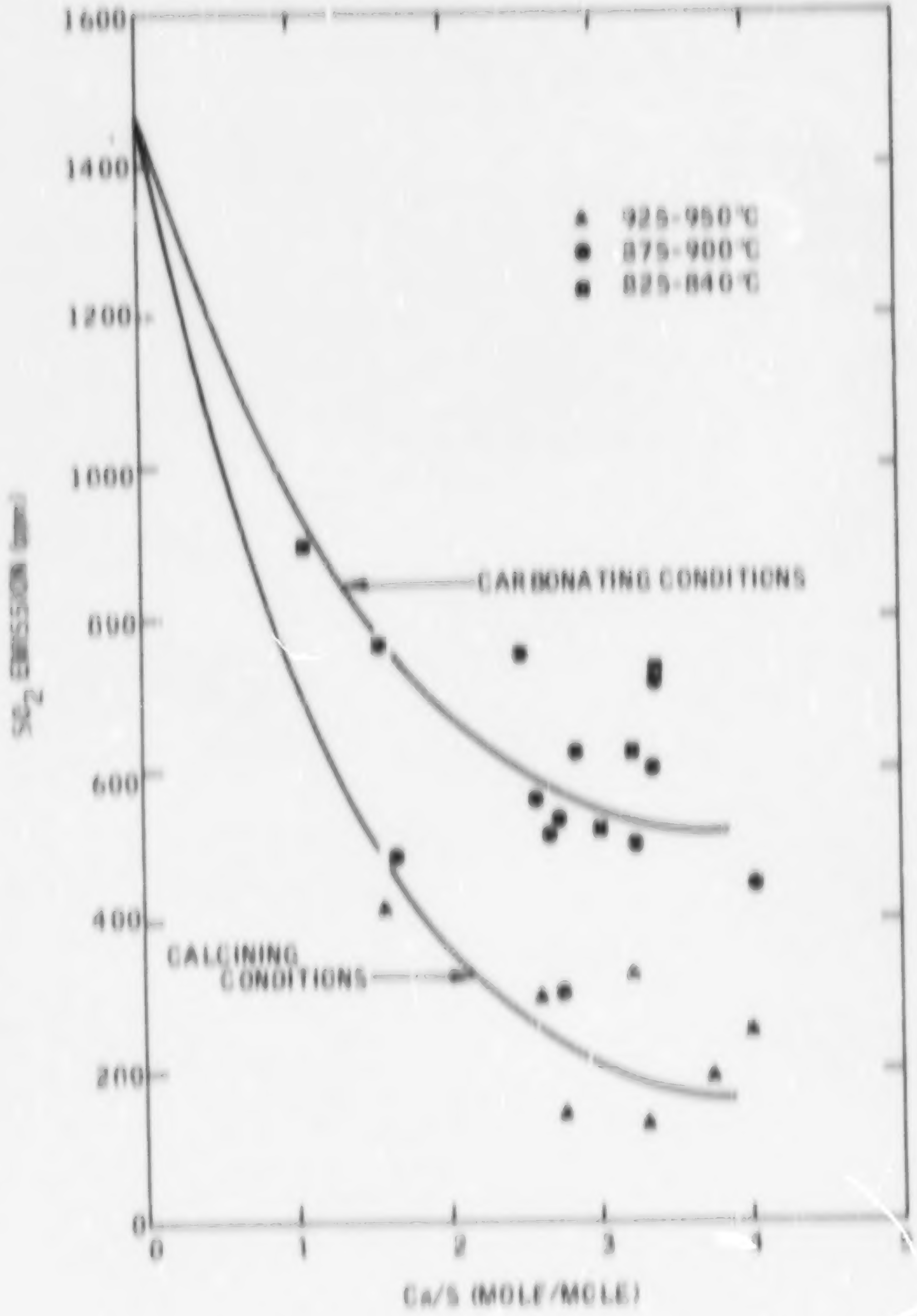
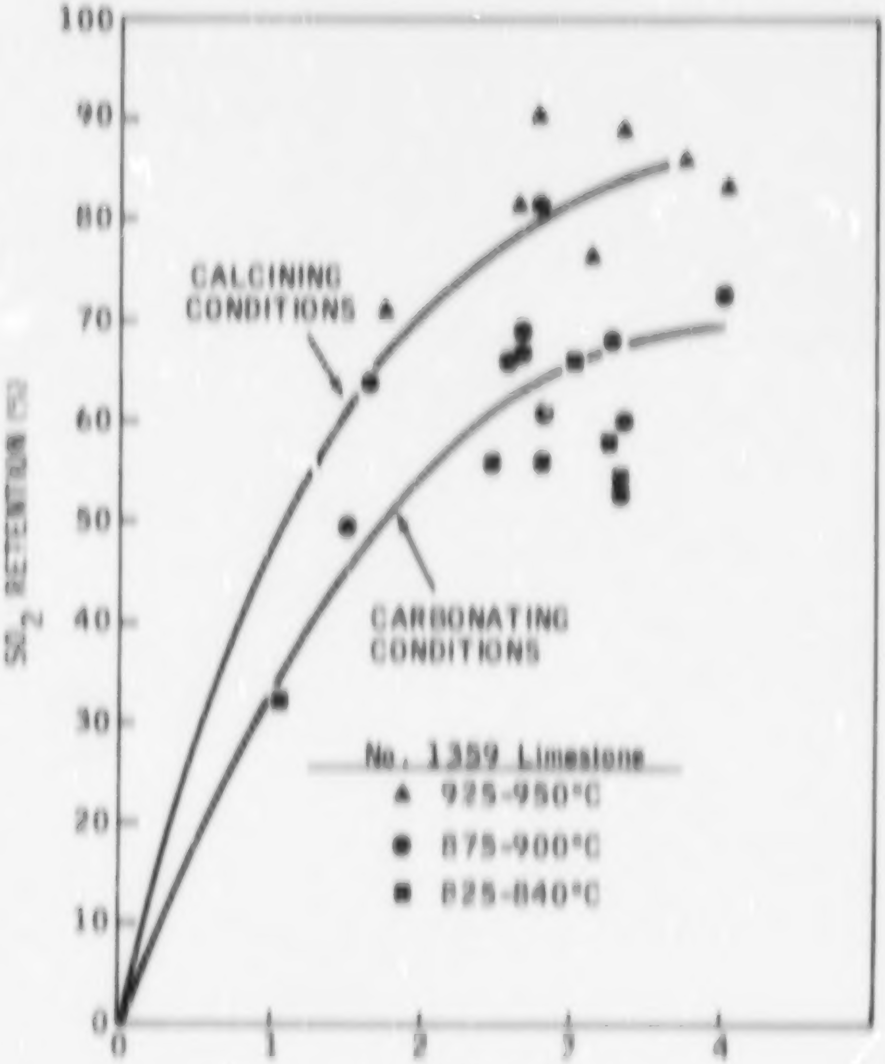


FIGURE III-18

$SO_2$ RETENTION VS. Ca/S RATIO FOR LIMESTONE NO. 1359

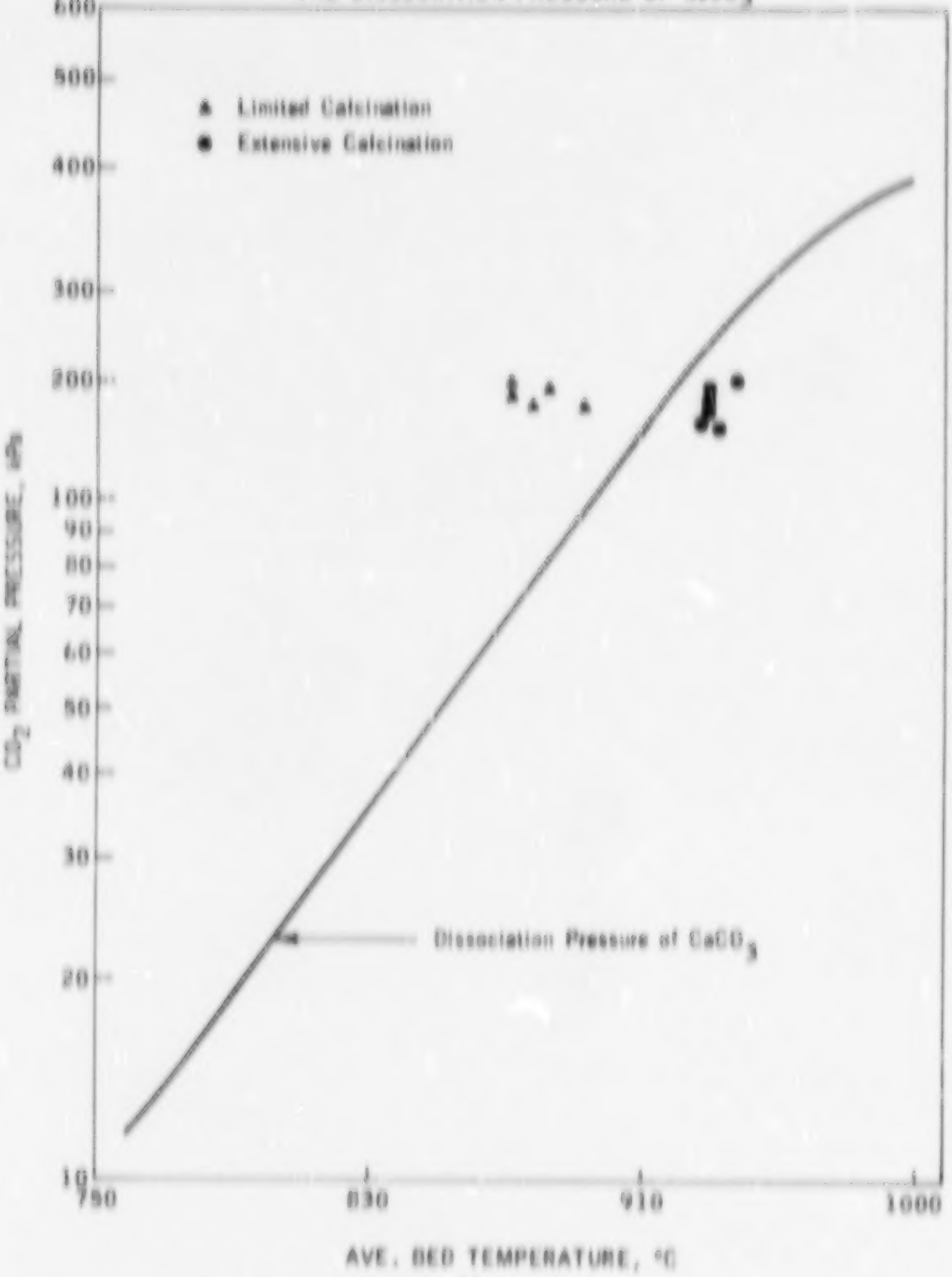


## TABLE III-7. UTILIZATION AND CALCINATION OF LIMESTONE NO. 1359

| Run No. | Temp. °C | Ca/S Mole/Mole | Bed Composition | | |
|---|---|---|---|---|---|
| | | | CaO Mole % | $CaCO_3$ Mole % | $CaSO_4$ Mole % |
| 19.4 | 880 | 2.65 | 12 | 62 | 26 |
| 19.5 | 890 | 2.7 | 11 | 65 | 25 |
| 19.6 | 820 | 3.2 | 5 | 77 | 18 |
| 19.7 | 940 | 4.0 | 77 | 1 | 21 |
| 19.9 | 950 | 2.56 | 64 | 4 | 32 |
| 20.2 | 950 | 3.75 | 76 | 1 | 23 |
| 26 | 885 | 2.75 | 29 | 37 | 33 |
| 29.1 | 875 | 2.8 | 8 | 72 | 20 |
| 30.1 | 885 | 1.5 | 3 | 64 | 33 |
| 30.2 | 930 | 3.3 | 72 | 1 | 27 |
| 30.3 | 885 | 1.6 | 9 | 52 | 39 |
| 30.4 | 835 | 1.1 | 3 | 67 | 30 |
| 31 | 830 | 3.0 | 29 | 49 | 22 |
| 32.1 | 950 | 3.2 | 72 | 5 | 24 |

FIGURE III-19

COMPARISON OF THE DEGREE OF SORBENT CALCINATION WITH THE DISSOCIATION PRESSURE OF $CaCO_3$



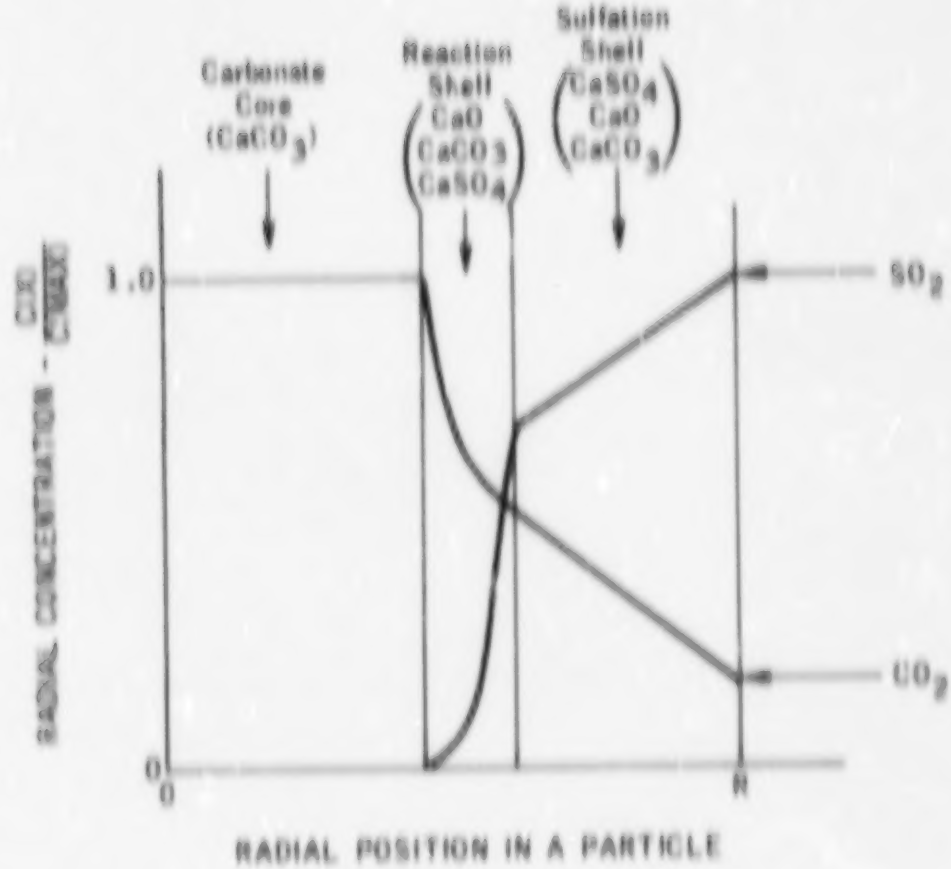

Carbonate Core ($CaCO_3$)
Reaction Shell ($CaO$ $CaCO_3$ $CaSO_4$)
Sulfation Shell ($CaSO_4$ $CaO$ $CaCO_3$)
1.0
0
0
R
$SO_2$
$CO_2$
RADIAL CONCENTRATION - $\frac{C(X)}{C\ MAX}$
RADIAL POSITION IN A PARTICLE

FIGURE III-20

MODEL FOR THE CALCINATION AND SULFATION REACTIONS IN A CALCINING ENVIRONMENT

Data obtained under both calcining and carbonating conditions can be correlated using an empirically modified form of the Ca/S ratio which accounts for the different calcination levels. If the calcium carbonate is considered to be an inert component, the effective Ca/S ratio is given by:

$$\text{Effective Ca/S} = \text{Ca/S} \cdot X_{(CaO + CaSO_4)}$$

where $X_{(CaO + CaSO_4)}$ is the mole fraction of the calcium present as the oxide or sulfate. The correlation is given in Figure III-21. As seen, all data are well correlated by a single line, irrespective of the operating temperature.

Effect of Limestone Utilization on the Rate of Sulfation--

The sulfation reaction first order rate constant can be calculated with experimental sulfur retention levels using the equation:

$$\text{First order rate constant, k, } (s^{-1}) = -\frac{1}{t_{gas}} \ln\left(\frac{100-\text{Retention}}{100}\right)$$

$$\text{where } t_{gas} = \frac{\text{Expanded Bed Height (m)}}{\text{Superficial Gas Velocity (m/s)}}$$

The dependence of the rate constant on the utilization is shown in Figure III-22. The activity decreases rapidly as sulfation occurs and becomes negligible well before the stone is completely reacted. Utilizations ranged between only 18 and 33% as compared to the sulfation level of 45-50% which appears to be the maximum attainable in a fluidized bed combustor. Limestone has an activity under calcining conditions that is three times greater than that under carbonating conditions.

Effect of Other Variables--

Pressure--Runs 31 and 32.1 were made at reduced pressures of 520 kPa and 600 kPa, respectively. The $SO_2$ emissions from these runs are compared to emission levels obtained from a correlation of data obtained at 930 kPa pressure in Table III-8. The agreement is good enough to show that the pressure effect is small or negligible.

Particle Size--In Runs 39.3 and 39.4, a limestone feed stream with a size range of 8-14 mesh was used in the first test while a 14-25 mesh feed was used in the other test. The mass average size ratio of the two feed streams was approximately 2. The sulfur retention levels were 68.7 with the fine feed and 70.3 with the coarse feed, a difference which is not significant.

## Operation at "Turndown" Conditions

The commercial application of pressurized fluid bed combustion (PFBC) would require that the system be operated under "turndown" conditions. To check the operability and sulfur removal capacity of PFBC under such conditions runs were carried out at nominally, 700° and 690°C using Pfizer dolomite and Grove limestone as sorbents. Results are shown in Table III-9.

FIGURE III-21

SULFUR RETENTION VS. EFFECTIVE Ca/S RATIO WHICH ACCOUNTS FOR DIFFERENT LIMESTONE CALCINATION LEVELS

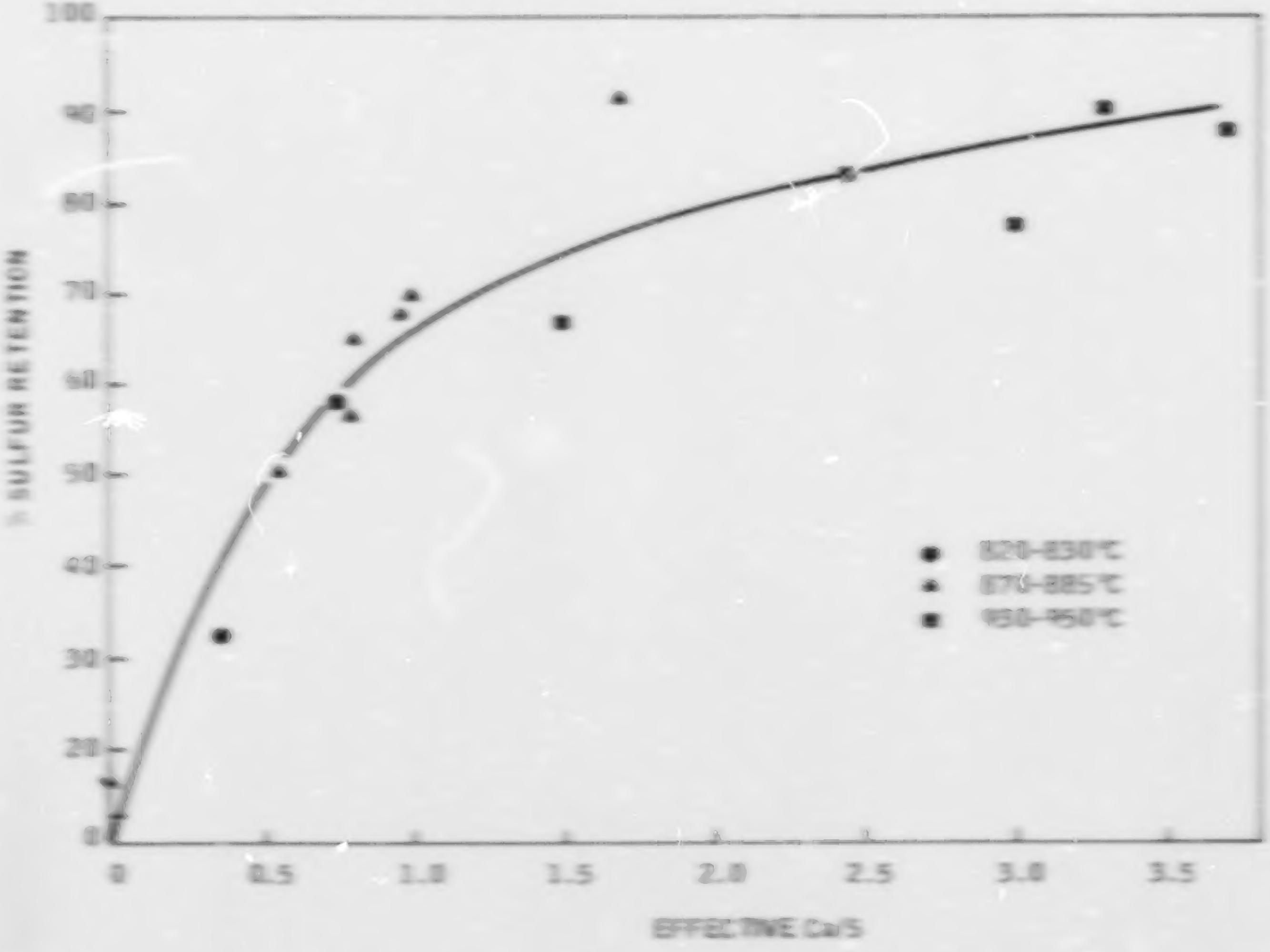


FIGURE III-22

ACTIVITY VS. CALCIUM UTILIZATION FOR LIMESTONE NO. 1359

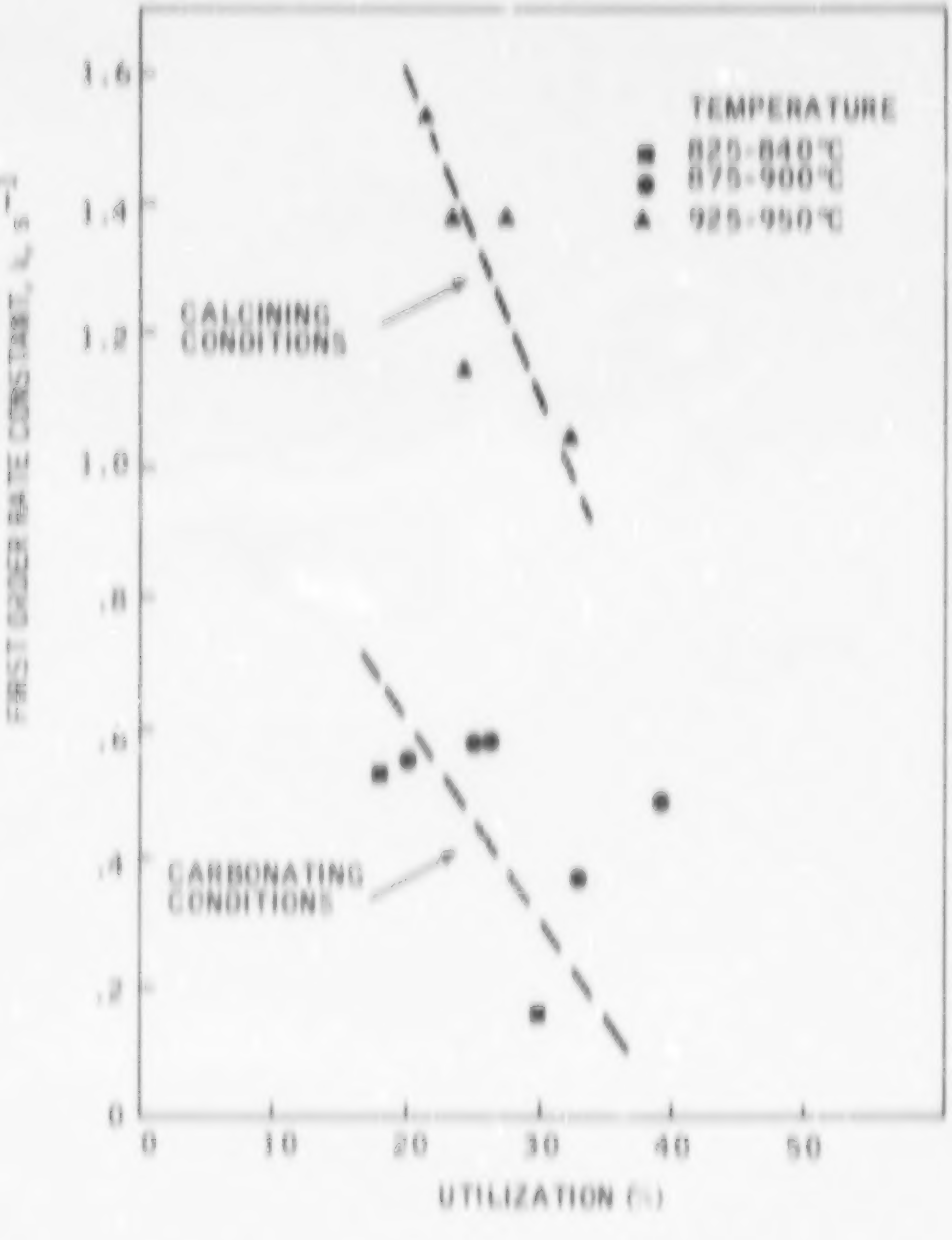


TABLE III-8. COMPARISON OF SULFUR RETENTIONS AT 930 kPa AND 600 kPa WITH LIMESTONE NO. 1359

| Run No. | Ca/S (Mole/Mole) | Temp. (°C) | Pressure (kPa) | Sulfur Retention (%) |
|---|---|---|---|---|
| 31 | 3.0 | 835 | 520 | 66 |
| * | 3.0 | 835-880 | 930 | 66 |
| 32.1 | 3.2 | 950 | 600 | 76 |
| * | 3.2 | 950 | 930 | 83 |

---

* Obtained from a correlation of $SO_2$ emissions for Grove limestone given in Figure

TABLE 11. - RESULTS OF RUNS AT STANDARD CONDITIONS

| Operating Conditions: | 38.1 | 38.3 | 38.4 | 38.5 | 38.6 | 39.1 | 39.2 | 39.3 |
|---|---|---|---|---|---|---|---|---|
| Avg. Bed Temperature, °C | 891 | 762 | 762 | 690 | 684 | 750 | 674 | 938 |
| Ca/S Molar Feed Ratio | 0.61 | 0.94 | 1.1 | 1.5 | 1.4 | 2.5 | 2.5 | 3.5 |
| Sorbent | PD | PD | PD | PD | PD | GL | GL | GL |
| Flue Gas Emission: | | | | | | | | |
| $SO_2$, ppm | 761 | 390 | 223 | 180 | 163 | 912 | 900 | 329 |
| $NO_x$, ppm | 135 | 125 | 149 | 114 | 102 | 120 | 174 | 125 |
| CO, ppm | 175 | 325 | 375 | 800 | 400 | 350 | — | 200 |
| Sulfur Retention, % | 40 | 48 | 71 | 76 | 82 | 0 | 9 | 70.3 |
| lb $SO_2$/M BTU | 2.29 | 1.42 | 0.78 | 0.64 | 0.5 | 2.69 | 2.86 | .80 |

---

PD = Pfizer Dolomite (No. 1337)
GL = Grove Limestone (No. 1359)

Operations were very good during these tests; temperature control was excellent and combustion was sufficient to give reasonable carbon monoxide emissions. Near the end of the runs with dolomite, the temperature was reduced further to determine the lowest feasible operating temperature. At 800°C, the temperature control was becoming erratic and carbon monoxide emissions were much higher so no further reduction in temperature was attempted.

As indicated in Table III-9, the sulfur retention was maintained under urndown conditions using dolomite as absorbent but retention was very poor t the lower temperatures when limestone was used. The poor results with imestone are due to inadequate calcination at the lower temperatures. hese results could have a strong influence on the choice of dolomite vs. limestone in a commercial unit. The use of precalcined limestone may permit its use under turndown conditions. This will be studied in the future.

### Sorbent Feed Required to Meet the EPA $SO_2$ Emission Standard

A plot of sulfur retention vs. the sorbent mass feed rate (Figure III-23) shows that calcined Grove limestone (930°C combustion temperature) is far more effective than the partly calcined stone (880°C combustion temperature). It also shows that Pfizer dolomite is more effective on a weight basis than calcined limestone at sulfur retention levels greater than 70%. While far higher utilizations can be achieved with dolomite than with limestone, dolomite contains only ~50 weight % $CaCO_3$ as compared to a ~100% $CaCO_3$ content for limestone. Figure III-23 can be used to estimate the sorbent feed rate required to meet the EPA standard for any sulfur coal by calculating the equivalent retention level. While the data was acquired with a 2% sulfur coal, the sulfation reaction kinetics are approximately first order in $SO_2$, meaning that the conversion profiles should be independent of the initial $SO_2$ concentrations or coal sulfur content. Table III-10 giving the sorbent feed requirements for 2, 3, 4 and 5% sulfur coals with 32.5 kJ/g heating value was prepared in this manner. At a coal sulfur level of 2%, 20% less limestone is needed as compared to dolomite to obtain the required 90% retention level. The sorbents are equally effective for a 3% sulfur coal. At higher coal sulfur levels, dolomite becomes increasingly more attractive. Thirty percent more limestone than dolomite is required to meet the standard with a 5% sulfur coal. With this coal, the Ca/S molar feed ratios would be 3.25 for limestone and 1.9 for dolomite. These values form approximate upper bounds on the molar feed rate requirement, as 90% of the U.S. coal reserves have a sulfur content under 5%. The data given in Table III-10 were estimated for a gas phase residence time of 3 s.

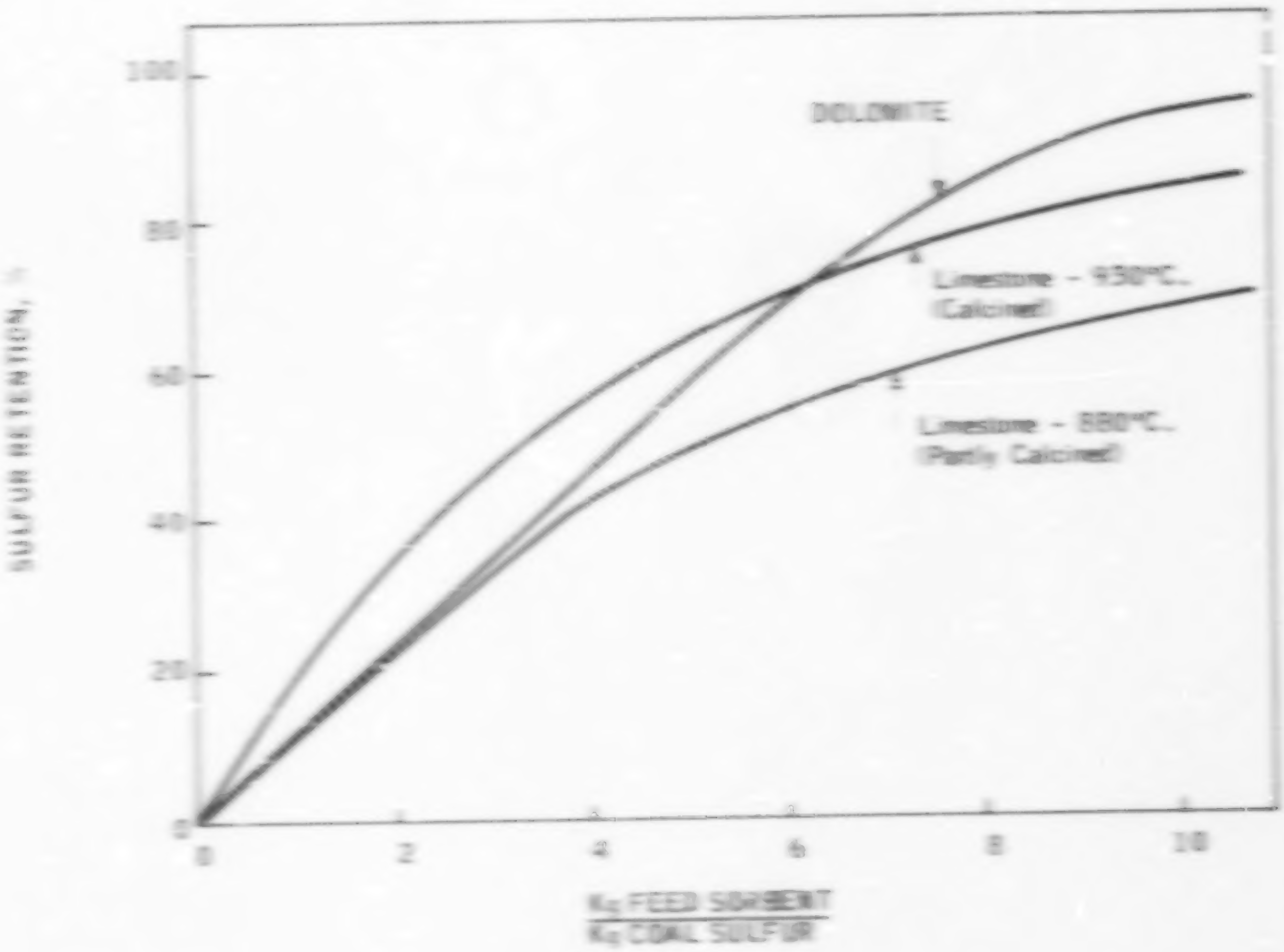
### Determination of the Accuracy of the $SO_2$ Emission Data

Inert Bed Runs--

As shown in Table III-11, excellent sulfur balances were calculated for the five test conditions of the inert bed run series, 28.1-28.5. The balances average 103%. Only 2.5% of the sulfur fed with the coal was retained by the ash, the bulk of the sulfur appeared as $SO_2$ in the flue gas. The bed sulfur analysis was 3.1%, higher than anticipated given the low surface area of the alumina. Approximately 10% of the total sulfur feed was absorbed by the bed. Almost certainly, much of the sulfur retention occurred during the initial stages of the run and did not significantly affect the sulfur balances, which are for a 2 to 3 hour period of steady state operation.

FIGURE III-23

COMPARISON OF DOLOMITE NO. 1337 AND LIMESTONE NO. 1359
AS $SO_2$ SORBENTS ON A MASS FEED RATE BASIS



## TABLE III-10. SORBENT REQUIREMENT TO MEET THE EPA $SO_2$ EMISSION STANDARD (3)

| Coal Sulfur (%) | Retention Level Req'd (%) (1) | Ca/S (Mole/Mole) Limestone(2) | Ca/S (Mole/Mole) Dolomite | kg Sorbent/100 kg Coal Limestone(2) | kg Sorbent/100 kg Coal Dolomite |
|---|---|---|---|---|---|
| 2.0 | 59 | 1.3 | 0.8 | 8.3 | 10 |
| 3.0 | 71 | 2.1 | 1.0 | 20 | 20 |
| 4.0 | 79 | 2.8 | 1.2 | 36 | 29 |
| 5.0 | 84 | 3.2 | 1.3 | 51 | 40 |

----------

(1) For coal with 32.5 kJ/g (14,000 BTU/lb) higher heating value.
(2) Calcining Conditions
(3) Estimates are for 3 s gas residence time.

## TABLE III-11. SULFUR BALANCES - INERT BED RUN SERIES

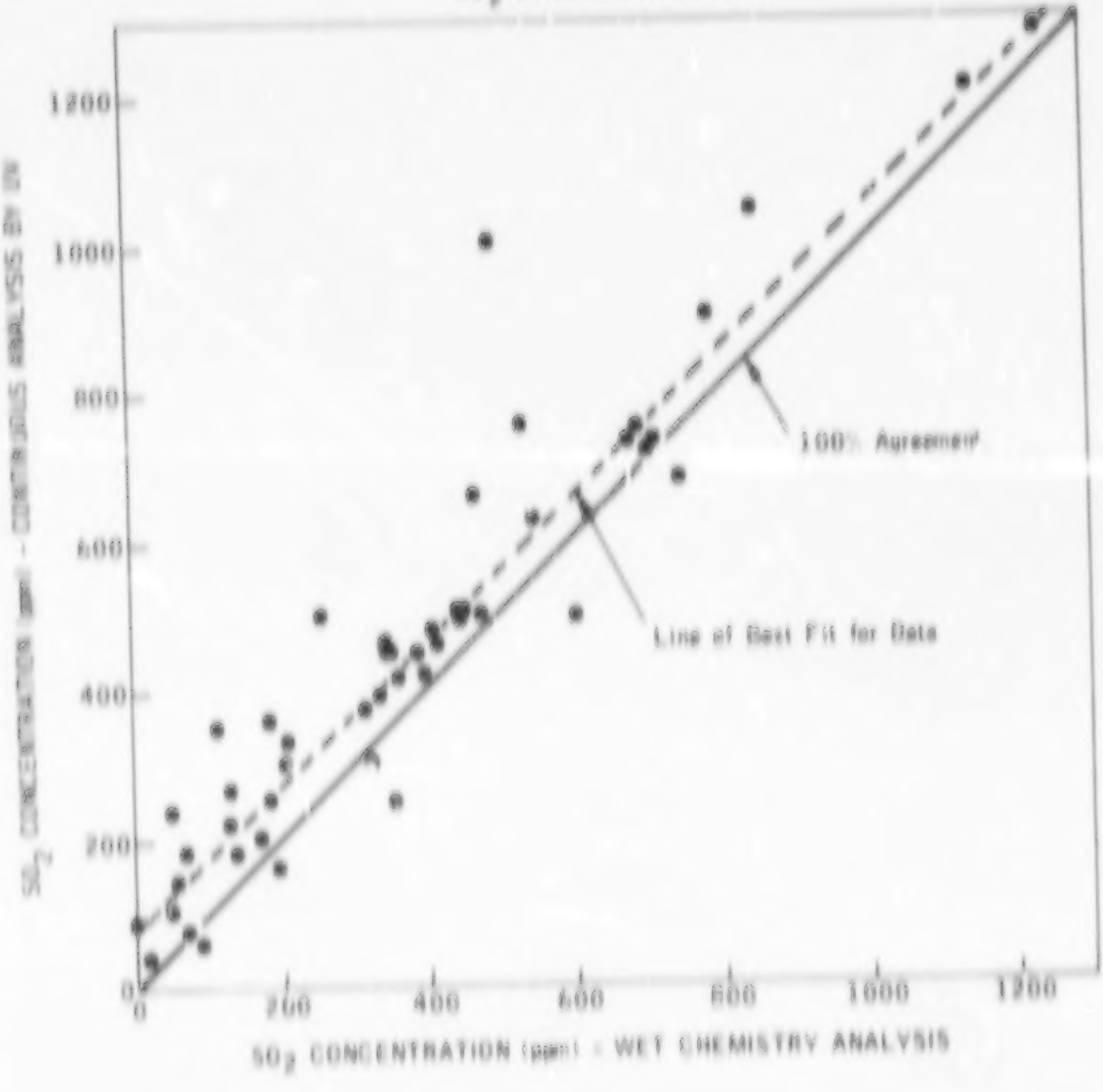
| Run No. | $\frac{\text{kg Sulfur out}}{\text{kg Sulfur in}} \times 100$ |
|---|---|
| 28.1 | 103 |
| 28.2 | 102 |
| 28.3 | 100 |
| 28.4 | 107 |
| 28.5 | 101 |
| | Avg. 103 ± 3 (1σ) |

Wet Chemistry $SO_2$ Determinations--

The $SO_2$ concentration in the flue gas was determined with a wet chemistry technique during many of the runs. The purpose was to verify the accuracy of the monitored $SO_2$ emissions. In most instances, the sample analyzed was drawn from a position a short distance downstream of the flue gas sampling port, before the gas had contacted the filter, pressure regulator or dryer used to prepare the flue gas for the continuous analyzers. The gas temperature at the sampling points was 110 to 120°C. In earlier runs, the sample was not filtered. In later runs, a glass fiber filter was installed on the sample line. No significant effect on $SO_2$ measurements was seen. The sample was passed from a heated line to a series of bubblers containing an isopropyl alcohol solution to absorb $SO_3$, and a hydrogen peroxide solution to absorb the $SO_2$. The amounts of $SO_2$ absorbed in the hydrogen peroxide solution were determined titrimetrically using sodium hydroxide as the titrant and methyl orange as the indicator. Four determinations on $SO_2$ calibration gases gave an agreement of 100 ± 5 (1σ) %.

The results are listed in Appendix Table C and the agreement between the monitored (UV) and wet chemistry $SO_2$ concentrations is examined in Figure III-24. The line of best fit for the data is offset by about 70 ppm from the line of 100% agreement with the monitored values higher. This implies that either a flue gas constituent was causing interference with the Dupont UV $SO_2$ analyzer readings or that the wet chemistry determinations were low. To determine

FIGURE III-24
COMPARISON OF MONITORED AND WET CHEMISTRY
$SO_2$ CONCENTRATIONS



which was the case, sampling for wet chemistry analysis was performed both at the sampling port on the miniplant and in the control room from a point very close to and the instruments during runs 34-39.4. The ratio between the wet chemistry and monitored values was 98 ± 8 (1σ) % for the determinations made just prior to the instruments while the ratio was only 60 ± 22 (1σ) % for those determinations made at the sampling port. The discrepancy appears to be with the wet chemistry determinations, which may be affected by the samples' high dew point. The agreement is adequate, however, to give confidence to the monitored $SO_2$ concentrations.

## $SO_3$ Emissions

$SO_3$ emissions in the flue gas were measured using a wet chemistry method. $SO_3$ was absorbed in an isopropyl alcohol solution which was titrated with barium perchlorate to a thorin end point. Results are given in Appendix Table C.

A total of *22* $SO_3$ determinations made during runs 19.2-26 had an average value of 5.6 ± 6 (1σ) ppm. The 22 $SO_3$ determinations made during runs 27.2-27.21 had a higher average value of 23 ± 15 (1σ) ppm. The reason for the higher $SO_3$ concentrations, whether process dependent or experimental, is not known. Subsequent determination of $SO_3$ concentrations in runs 34-39 again gave low values. Further work will be required to determine the level of $SO_3$ emissions.

## Nitrogen Oxide Emissions

Nitrogen oxide emissions ranged between 50 and 209 ppm or 0.04 to 0.17g (as $NO_2$)/MJ (0.1 to 0.4 lbs/M Btu). The data are shown in Figure III-25 where $NO_x$ emissions are plotted against percent excess air. Though the operation conditions varied greatly in runs 19.2-39.4, the only statistically significant variables were the excess air (or flue gas oxygen concentration) and bed temperature. The $NO_x$ emissions increased about 4 fold, from 0.04 to 0.17 g/MJ over the 5-100% range of excess air. The temperature effect in the 680-950°C range was secondary and caused only a 25% increase in the emission level. The emissions are well below the EPA standards of 0.3 g (as $NO_2$)/MJ (0.7 lbs/M BTU) and have an average value of only 0.09 g/MJ (0.2 lb/M BTU) at 15% excess air.

The data from run 34, shown in Figure III-26, suggest that in some circumstances the sulfation level of the bed and/or $SO_2$ emission level affect the $NO_x$ emission level. When the Ca/S mole ratio of the Pfizer dolomite/coal feed was increased from 0.75 to 1.5, the $SO_2$ emissions dropped from 300 ppm to 0 ppm (steady state conditions were not reached) and the NO emissions increased from 100 ppm to 200 ppm. The NO emission increased slightly more to 225 ppm when the Ca/S ratio was increased to 2.5. Use of a sulfated dolomite feed stream during the last four hours of the run caused the NO emissions to drop to 65 ppm, while the $SO_2$ emissions rose to 60 ppm.

## CO Emissions

CO emissions were independent of the bed temperature between 825 and 950°C and ranged from 70-225 ppm. At turndown temperatures of 680°C and 760°C, the emissions were 4 to 5 times higher. The emissions are plotted

FIGURE III-25

$NO_x$ EMISSIONS VS. EXCESS AIR

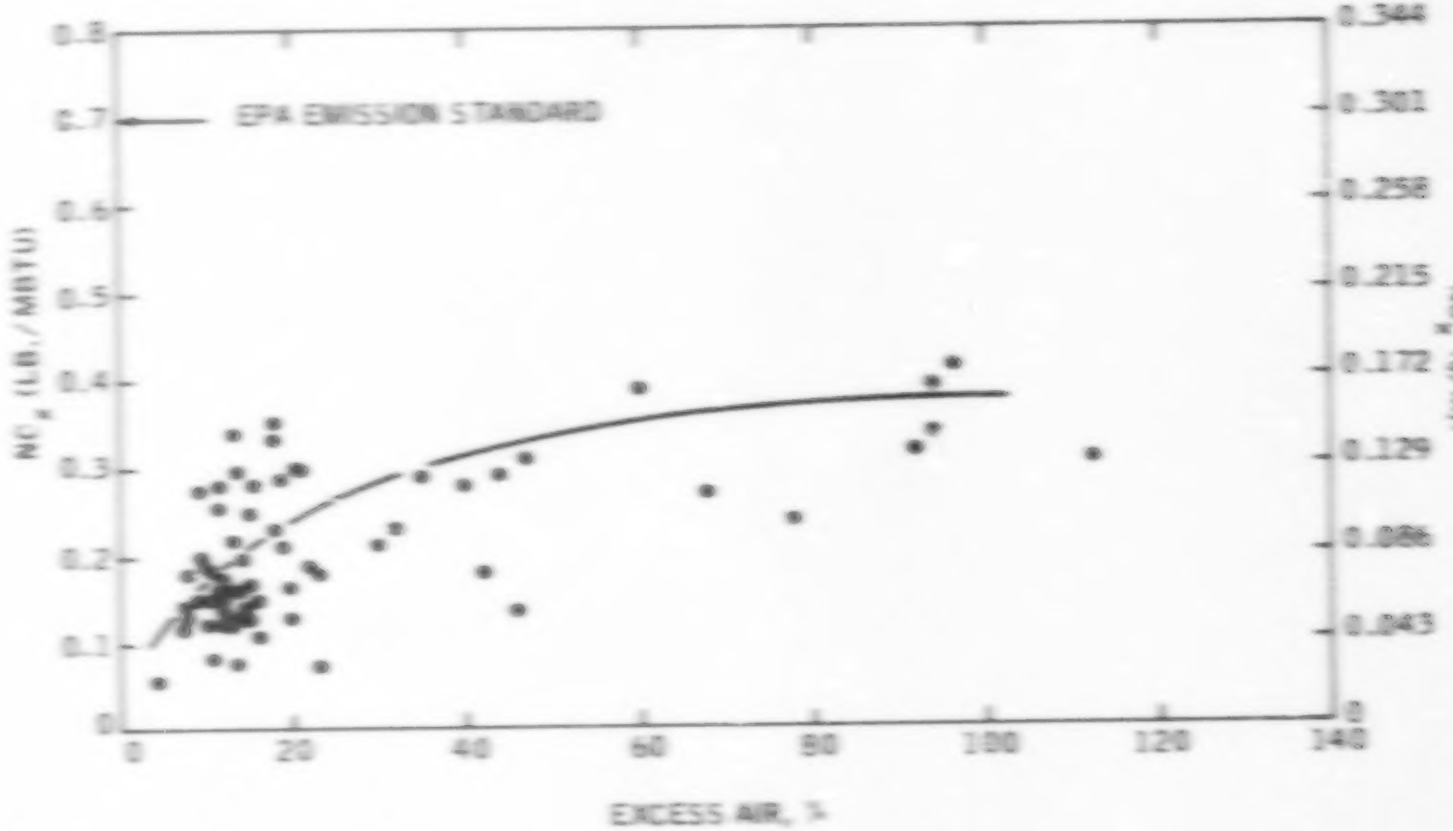
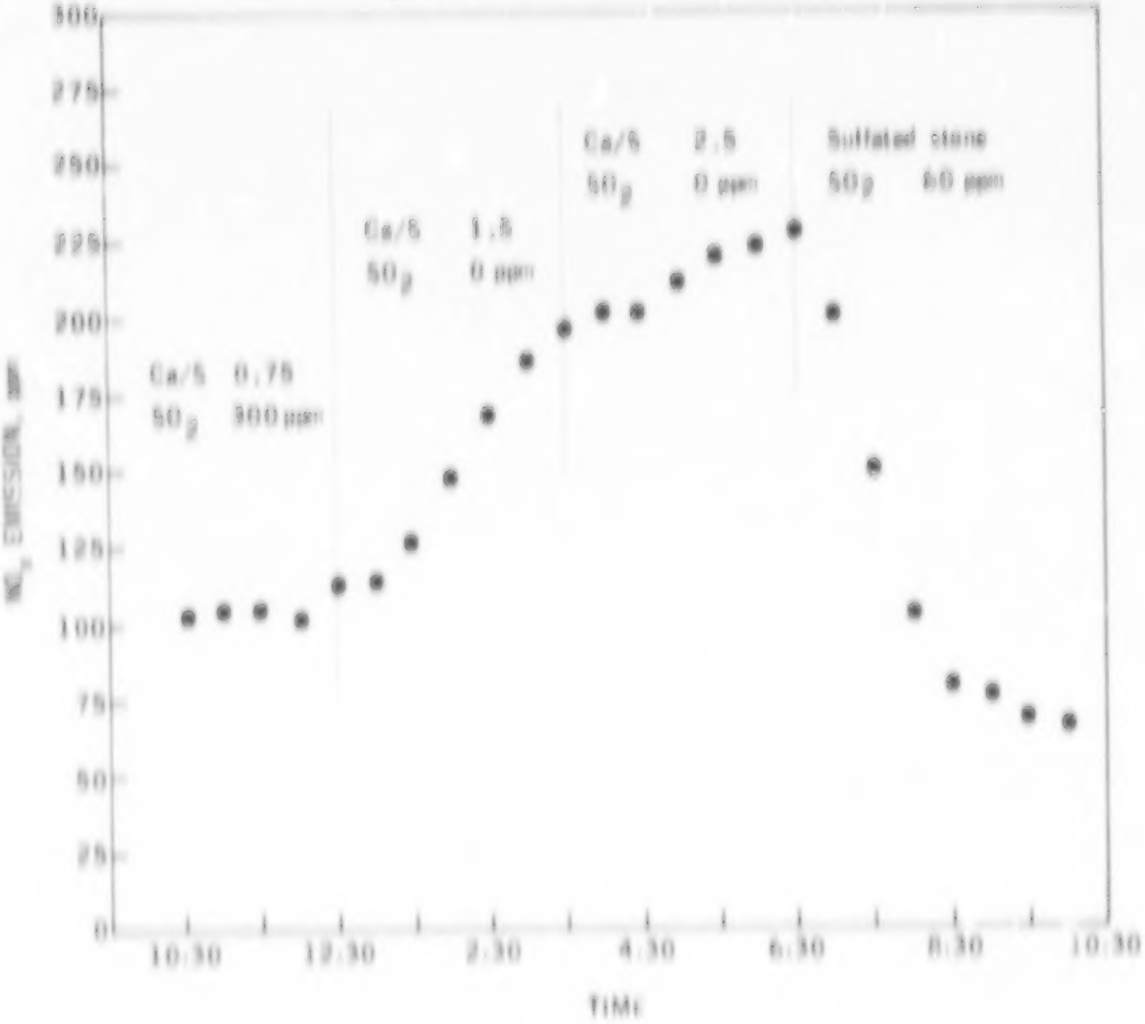


FIGURE III-26

NO$_x$ EMISSIONS DATA - RUN No. 34



in Figure III-27. The heat losses due to incomplete combustion of CO to $CO_2$ were low, and ranged from 0.05-1.0% at higher temperatures to 0.6% at turndown temperatures as shown in Table III-12. CO emissions were independent of the sorbent used.

TABLE III-12. HEAT LOSS DUE TO INCOMPLETE COMBUSTION OF CO TO $CO_2$

| Run No. | Temperature (°C) | Heat Loss as Percent of Total Heat Input |
|---|---|---|
| 38.5 | 690 | 0.59 |
| 38.6 | 680 | 0.25 |
| 38.4 | 760 | 0.27 |
| 38.3 | 760 | 0.20 |
| 39.1 | 760 | 0.21 |
| Remaining Runs | 825 - 960 | 0.05 - 0.10 |

## Particulate Emissions

Solid matter is emitted from the miniplant combustor from several points. The major source of solid matter is used sorbent removed from the combustor. This material consists largely of sulfated sorbent with a smaller quantity of coal ash and char. The average size is usually only slightly less than that of the fresh sorbent. Most of the coal ash and some used sorbent fines are entrained (or elutriated) from the combustor by the flue gas and enter the flue gas particulate removal system. Currently, this consists of two cyclones operated in series. The first cyclone captures the large particulates and returns them to the combustor to increase the carbon combustion efficiency. The amount and particle size of the particulates entering the first stage cyclone and the particulates recycled to the combustor cannot, at the present time, be measured. The particulates leaving the first stage cyclone with the flue gas consist of flyash and some attrited sorbent fines. This particulate stream then enters the second stage cyclone and the portion of it which is retained by the second stage cyclone is routinely measured, analyzed and sized. Flue gas leaving the second stage cyclone is sampled through an isokinetic sampling system and the particulate loading, size distribution and composition are determined.

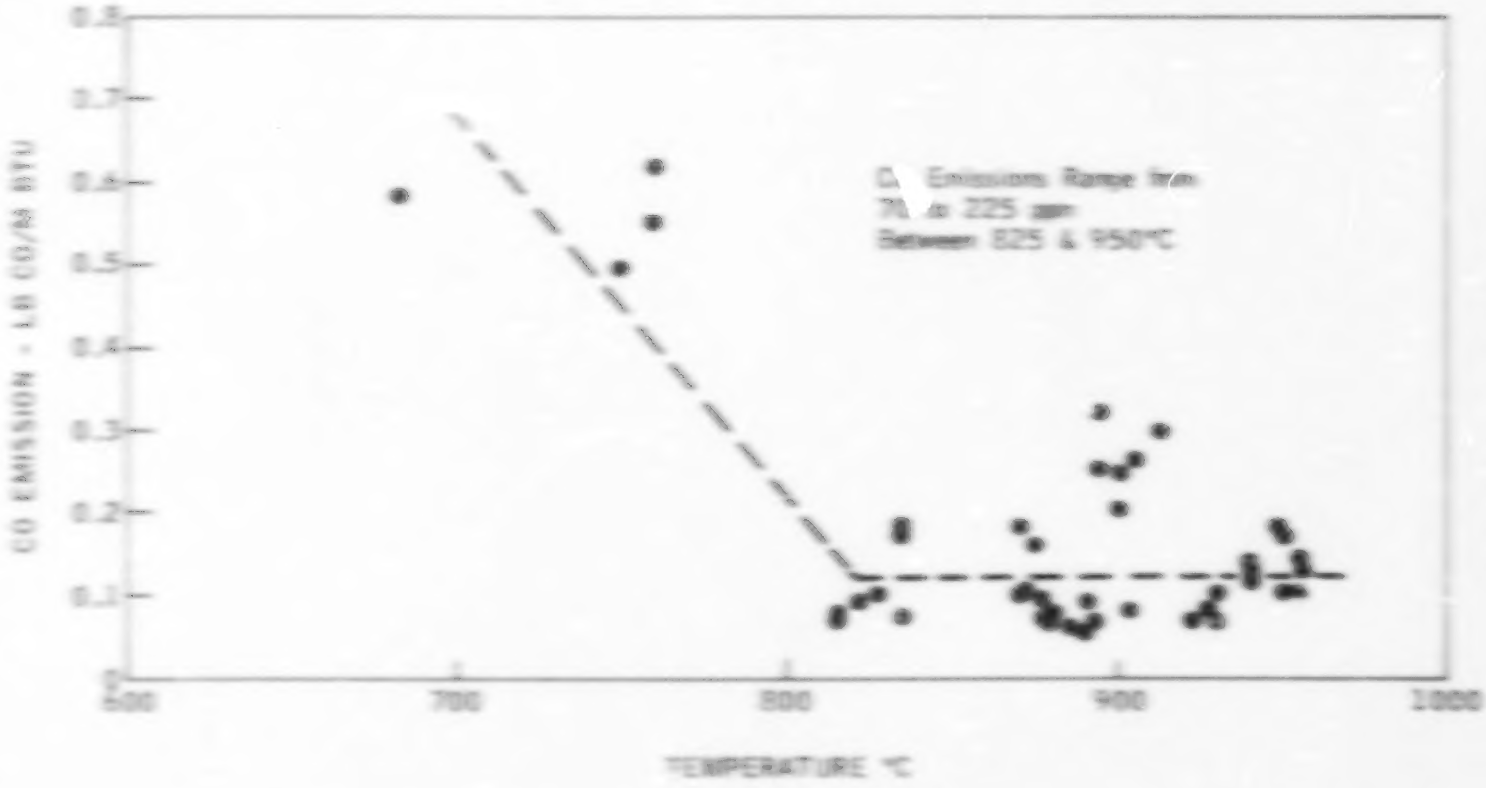
In the following sections, these particulate emissions are discussed.

### Sorbent Elutriation Rates--

Elutriation rates for Grove No. 1337 limestone and Pfizer No. 1359 dolomite were calculated from the quantity of calcium in the flyash collected by the second cyclone. The flyash sample analyzed from runs prior to 27.1 were blended to be representative of the entire run. This was done by mixing the samples of the different lockhopper dumps in the proper proportion. In later runs, individual dumps collected over 1 to 3 hour intervals of steady state operation were analyzed to obtain data which would be more representative of the condition tested. Sorbent losses to a large extent, represent the elutriation of the fines formed by attrition. Most bed particles coarser than 100 microns are captured by the first cyclone and recycled.

FIGURE III-27

CARBON MONOXIDE EMISSION VS. TEMPERATURE



The elutriation rates for both calcined and uncalcined Grove limestone were low, as shown in Table III-13. Detailed data are given in Appendix Tables D, E and F. Over the range of Ca/S feed rates used, 1.45-4.0, and ranges of superficial gas velocities, 1.4-2.2 m/s, the elutriation rate represented ~10% of the sorbent feed. The limestone feed rate required to match the loss was equivalent to a Ca/S feed ratio of about 0.24. The elutriation rates for calcined and uncalcined limestone were not significantly different. Data from runs 19.7, 20.1 and 21 were higher than the average. The reason is not known. Rates measured at low temperatures, 680 to 750°C suggest the attrition and elutriation rates are lower in this temperature range (see Appendix Table D).

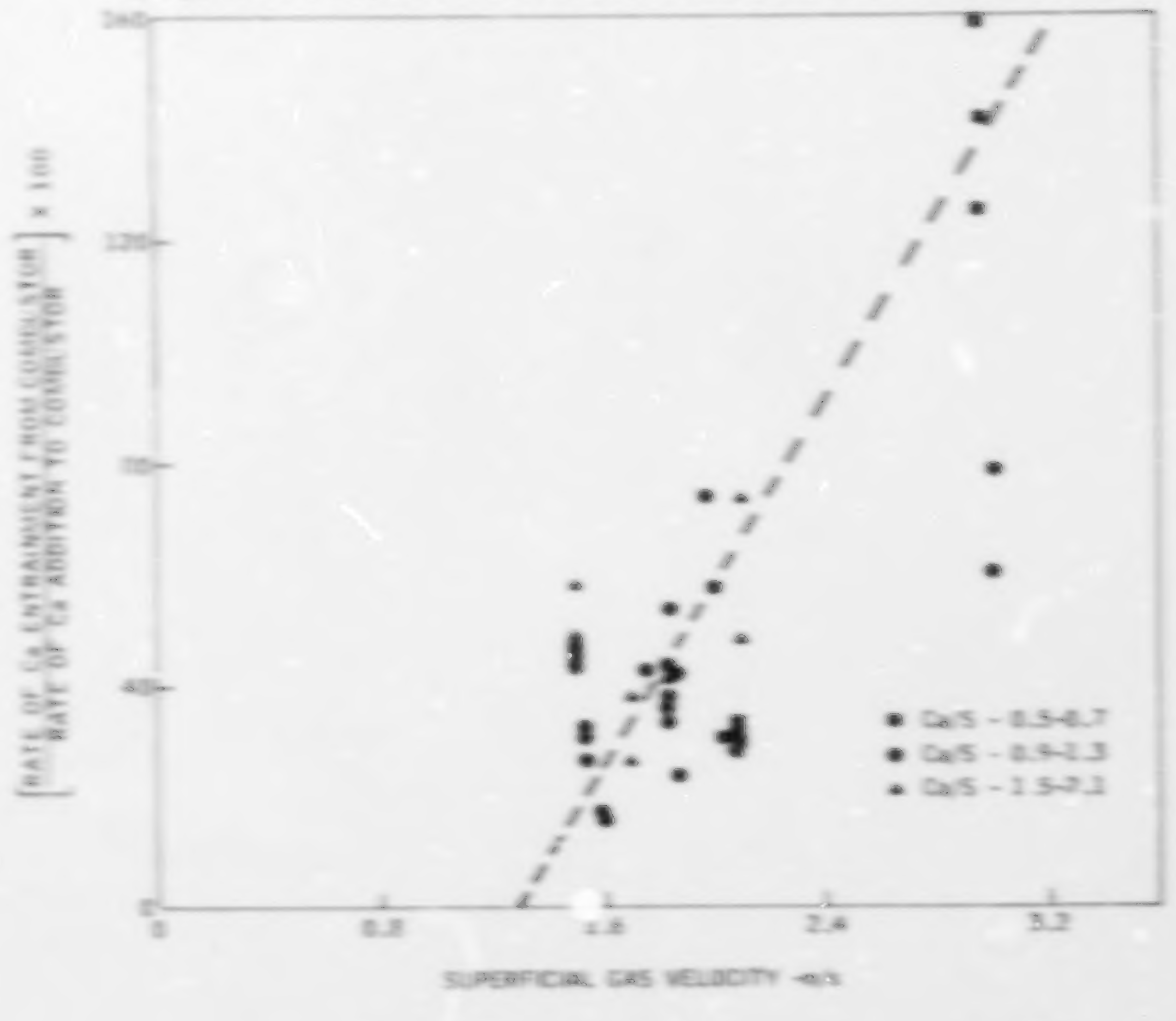
TABLE III-13. SORBENT ELUTRIATION LOSSES

| Sorbent | Sup. Vel. (m/s) | Feed Rate (Ca/S) | Elutriation Losses (Equiv. Ca/S) | (% of Feed) | (Vol. % Bed hr) |
|---|---|---|---|---|---|
| Limestone Grove 1359 | | | | | |
| - uncalcined | 1.4-2.2 | 1.5-2.8 | 0.2 | 12 | 1.1 |
| - calcined | 1.6-2.5 | 2.0-4.0 | 0.2 | 8 | 0.8 |
| Dolomite | | | | | |
| - low velocity | 1.5-2.1 | 0.6-1.8 | 0.4 | 38 | 2.1 |
| - high velocity | 3 | 0.5-1.3 | 0.8 | 110 | 5.5 |

Elutriation rates for dolomite, given in Table III-13, were substantially greater than those for limestone. Over a range of velocities from 1.5-2.1 m/s, the rates averaged 38% of the sorbent feed and the loss was equivalent to a Ca/S feed ratio of 0.4. At a velocity of 3 m/s, 110% of the sorbent feed was elutriated on the average and the Ca/S equivalent was 0.8. ANL (6) was able to correlate elutriation data well for Tymochtee dolomite by plotting the fraction of the feed calcium entrained from the combustor against the gas velocity. The fraction of the feed elutriated was found to be independent of the Ca/S ratio. This approach was used to correlate the data shown in Figure III-28. The elutriation rates for the miniplant also appear to be largely independent of the Ca/S ratio and to increase with gas velocity. There is much uncertainty in the slope of the line of best fit, due to the scatter in the data and to the lack of data at higher velocities. A comparison of the Argonne and miniplant correlations shows that the elutriation rates in the Argonne unit are higher. The primary reason for this is that in the miniplant, most of the fines are recycled by the first cyclone. In runs with dolomite, over one-half of the bed material had a terminal velocity lower than the superficial gas velocity and the bed contained particles as fine as 200 microns. The recycle rates must therefore, be substantial. The maximum feasible operating velocity, that velocity at which 100% of the feed is elutriated is 1.9 m/s in the Argonne unit. The maximum velocity in the miniplant has not been determined with accuracy, but is below 3 m/s.

FIGURE III-22

EFFECT OF GAS VELOCITY AND Ca/S RATIO ON ELUTRIATION OF DOLOMITE No. 1337



Particulate Emission Levels--

Particulate emission levels in the flue gas leaving the second stage cyclone were measured using the sampling system described earlier in this section. Data are shown in Table III-14 for runs 20 through 39. Over this period the sampling system appeared to be operating satisfactorily with deviations from isokinetic conditions generally less than ± 15%. Except for the earlier runs (through 30.1), particulate levels ranged from 0.9 to 4.8 g/m³ (0.4 to 2.1 gr/SCF) and averaged 2.5 g/m³ (1.1 gr/SCF). The spread in the measured values is greater than expected and could be due to experimental problems with the sampling system or variable performance of the second stage cyclone.

The particulates captured in the second stage cyclone represent a concentration in the flue gas of 7 to 18 g/m³ (3 to 8 gr/SCF). Therefore, the concentration of particulates entering the second stage cyclone, obtained by summing the amount captured in the cyclone and the concentration measured in the flue gas leaving the cyclone is in the range of 7 to 23 g/m³ (3 to 10 gr/SCF).

Particulate Size Distribution--

Size distributions were measured for the spent sorbent removed from the combustor during a run, the material retained in the second stage cyclone, and the fine particulates captured in the flue gas particulate sampling system.

Spent Sorbent--The spent sorbent removed from the combustor during the course of a run and the sorbent remaining in the combustor after a run was completed were sized by sieve analyses. The results are given in Table III-15 and are compared to the size distribution of the sorbent fed to the combustor during the runs. Detailed data are given in Appendix Tables G and H. As seen in Table III-15, the spent sorbent is significantly reduced in size, showing the effects of particle attrition and recycle of the first cyclone capture back to the combustor. If particles were not recycled from the first cyclone, an increase in the mass median size of the spent sorbent would be expected due to the loss by entrainment of the smaller particles. It can also be seen that spent limestone sorbent is significantly coarser than spent dolomite, reflecting the higher attrition rate of dolomite. The spent sorbent removed from the combustor during a run is also coarser than the material sampled from the bed after a run has been completed. The reason for this result is not known, but may be due to particle size segregation within the combustor. The spent sorbent is removed from a port in the combustor wall and it is possible that coarser particles may be gathering at the wall. This could be due to the solids circulation patterns within the combustor which are believed to be upward in the center and downward at the wall. Smaller particles carried up the center of the combustor may be entrained into the cyclone system, while the larger particles are disengaged in the freeboard section of the combustor and fall down near the wall where they can be preferentially removed through the solids removal port.

It can also be seen that the particle size distribution data show large variances from run to run as indicated by the standard deviations given in Table III-15. This could be caused by a number of operating factors such as differences in superficial velocity, bed depth, etc.

TABLE III-14. MINIPLANT FLUE GAS PARTICULATE SAMPLING SUMMARY

| Run No. | Sampling Time (hrs) | Total Solids Collected (g) | Solids Rate in Probe (g/min) | Solids Rate in Probe (gr/min) | Deviation from Isokinetic Sampling (%) | Solids Loading in Flue Gas (mg/m$^3$) | Solids Loading in Flue Gas (gr/SCF) |
|---|---|---|---|---|---|---|---|
| 28.1 | 2.70 | 26.9 | 0.17 | 2.56 | 0 | 590 | 0.26 |
| 28.4 | 1.30 | 4.0 | 0.05 | 0.77 | 0 | 370 | 0.16 |
| 28.5 | 1.25 | 7.0 | 0.09 | 1.44 | 0 | 300 | 0.13 |
| 29 | 2.58 | 19.0 | 0.12 | 1.86 | −26 | 530 | 0.23 |
| 30.1 | 2.58 | 14.0 | 0.09 | 1.40 | −22 | 430 | 0.19 |
| 31 | 2.00 | 45.0 | 0.38 | 5.86 | −7 | 2890 | 1.26 |
| 32.2 | 2.75 | 25.0 | 0.15 | 2.33 | +6 | 920 | 0.40 |
| 32.3 | 2.50 | 27.0 | 0.18 | 2.79 | −22 | 1650 | 0.72 |
| 34.1 | 5.47 | 95.0 | 0.29 | 4.49 | +21 | 1420 | 0.62 |
| 36 | 4.42 | 85.0 | 0.32 | 4.97 | +21 | 1580 | 0.69 |
| 36.2 | 2.50 | 77.0 | 0.50 | 7.71 | +20 | 2220 | 0.96 |
| 37 | 3.00 | 86.0 | 0.48 | 7.42 | −48 | 2360 | 1.03 |
| 39.1 | 2.5 | 175.5 | 1.17 | 18.08 | +3 | 4813 | 2.10 |
| 39.2 | 2.5 | 186.4 | 1.24 | 19.28 | +3 | 4813 | 2.10 |

TABLE III-15. SPENT SORBENT PARTICLE SIZE DISTRIBUTION

|  | Particle Size (µm) |  |  |
|---|---|---|---|
| Material | 10% Less Than | 50% Less Than | 90% Less Than |
| Sorbent Feed | 880 | 1640 | 2280 |
| Spent Limestone |  |  |  |
| Removed During Run | 820 ± 90 | 1360 ± 130 | 2160 ± 110 |
| From Combustor After Run | 630 ± 180 | 1140 ± 180 | 1980 ± 150 |
| Spent Dolomite |  |  |  |
| Removed During Run | 370 ± 180 | 1230 ± 300 | 1940 ± 390 |
| From Combustor After Run | 360 ± 230 | 890 ± 430 | 1820 ± 310 |

Solids retained in second stage cyclone (flyash)--The material retained in the second stage cyclone is a mixture of flyash, unburned carbon residue and attrited sorbent fines. Composition data are given in a later section. This material has passed through the first stage cyclone and is fairly fine. Table III-16 gives particle size distribution data.

TABLE III-16. PARTICLE SIZE DISTRIBUTION - FLYASH

|  | Particle Size (µm) |  |  |
|---|---|---|---|
| Number of Runs | 10% Less Than | 50% Less Than | 90% Less Than |
| 42 | 6 ± 2 | 23 ± 16 | 103 ± 69 |
| 29 | 5 ± 1 | 18 ± 3 | 66 ± 9 |
| 12 | 7 ± 3 | 30 ± 11 | 171 ± 67 |

As seen, if data from 42 runs are included, the particle size distribution is 6, 23 and 103 µm for the three points in the distribution. However, if data are rejected which fall outside the 2σ limits, the 29 remaining runs show a size distribution of 5, 18 and 66 µm for the 10, 50 and 90% points. It is then obvious that the data from the 42 runs represent two size distribution populations. Twelve of the remaining 13 runs were averaged and gave size distributions of 7, 30 and 171 µm corresponding to the 10, 50 and 90% points in the distribution. One run was rejected even from this population. The major differences between the two particle size populations is at the upper end of the distributions, 66 vs. 171 µm at the 90% point in the distribution. It is believed that the difference is due possibly to variable performance of the cyclones. Some minor modifications have been made to the cyclones and more recent data are typified by the smaller size distribution. Detailed data are given in Appendix Table 1.

Fine Particulates--Fine particulates sampled isokinetically from the flue gas were sized using a combination of wet sieving and a Coulter Counter. The average size for six runs are given in Table III-17. As seen, the size distributions are fairly uniform, and average 2, 7 and 32 μm at the 10, 50 and 90% points in the size distributions. More detailed data are given in Appendix Table J.

TABLE III-17. PARTICLE SIZE DISTRIBUTION - FINE FLUE GAS PARTICULATES

| | Particle Size (μm) | | |
|---|---|---|---|
| Run No. | 10% Less Than | 50% Less Than | 90% Less Than |
| 31 | 1.8 | 8.0 | ~50 |
| 32.2 | 2.0 | 5.8 | 34 |
| 32.3 | 2.3 | 5.9 | 32 |
| 33 | 3.3 | 7.6 | 36 |
| 36.2 | 2.5 | 7.5 | 30 |
| 37 | 2.0 | 6.3 | 30 |
| Avg. | 2.3 ± 0.5 | 6.9 ± 0.9 | 32 ± 9 |

Particulate Composition--

Typical particulate chemical analyses for the used bed, solids removed from the bed during the run, flyash collected in the second stage cyclone and particulates in the flue gas leaving the second stage cyclone are given in Table III-18. Table III-19 gives data for the same runs expressed as percent carbon, ash and sorbent and also gives the CaO, $CaCO_3$ and $CaSO_4$ content of the sorbent portions of the various solids. Runs were included which covered a range of sorbent types, Ca/S ratios and temperatures. Detailed data are given in Appendix Tables K and L.

In general, the spent solids removed from the combustor contain less than 1% combustible carbon except at very low temperatures where carbon contents may be as high as 3%. Ash content in the used bed material varies from 2 to 25% and the sorbent content varies from 75 to 95%.

The flyash collected in the second stage cyclone generally contains 3 to 20% carbon. However, it tends to be lower when dolomite sorbent is used because of the dilution effect of the larger amount of entrained dolomite fines in the flyash. In this case, carbon contents as low as 3 to 5% are seen, especially in runs made at the higher combustor temperatures. The ash content in the collected flyash is 65 to 80% when limestone sorbent is used compared to 40 to 60% for dolomite sorbent. The sorbent content in the flyash generally runs from 15 to 20% for limestone compared to 30 to 60% for dolomite.

The fine particulates filtered from the flue gas contain 3 to 9% carbon. In general, fine particulates from runs made with dolomite contain lower carbon concentrations, due in part at least, to the dilution effect of higher

TABLE III-18. TYPICAL PARTICULATE ANALYSES

| Run No. | Sorbent | Ca/S (m/m) | Temperature (°C) | Source | Ca | Mg | SO4 | CO2 | C |
|---|---|---|---|---|---|---|---|---|---|
| 39.2 | Limestone | 2.5 | 670 | Bed-End of Run | Sample Not Taken | | | | |
| | | | | Bed-Rejected Solids | 32 | — | 6.6 | 31 | 3.3 |
| | | | | Fly Ash | 6.2 | — | 7.4 | 1.1 | 5.8 |
| | | | | Fine Particulates | 3.9 | — | 12.9 | 0.2 | 4.1 |
| 31.1 | Limestone | 3.0 | 840 | Bed-End of Run | 28 | — | 25 | 28 | 0.1 |
| | | | | Bed-Rejected Solids | 33 | — | 22 | 17 | 0.5 |
| | | | | Fly Ash | 6.8 | — | 7.2 | 1.3 | 18.1 |
| | | | | Fine Particulates | 4.2 | — | 7.7 | 0.9 | 7.2 |
| 32.3 | Dolomite | 1.5 | 840 | Bed-End of Run | 29 | 9 | 29 | 15 | 0.1 |
| | | | | Bed-Rejected Solids | Sample Not Taken | | | | |
| | | | | Fly Ash | 9.8 | 3.5 | 11 | 1.7 | 12.2 |
| | | | | Fine Particulates | 11 | 2.2 | 8.1 | 0.6 | 2.4 |
| 36.2 | Dolomite | 1.3 | 900 | Bed-End of Run | 26 | 13 | 37 | 12 | N.D.(1) |
| | | | | Bed-Rejected Solids | Sample Not Taken | | | | |
| | | | | Fly Ash | 16 | 8.9 | 21 | 1.8 | 2.8 |
| | | | | Fine Particulates | 6.2 | 3.6 | 9.5 | 0.6 | 3.6 |
| 37 | Dolomite | 0.5 | 900 | Bed-End of Run | 22 | 10 | 46 | 2.3 | N.D.(1) |
| | | | | Bed-Rejected Solids | Sample Not Taken | | | | |
| | | | | Fly Ash | 12 | 7.3 | 23 | 0.8 | 2.8 |
| | | | | Fine Particulates | 4.6 | 2.8 | 7.0 | 0.2 | 3.6 |

---

(1) N.D. - Not Determined

TABLE III-28. TYPICAL PARTICULATE COMPOSITION

| Run No. | Sorbent | Ca/S (m/m) Ca | Temperature (°C) | Source | – Composition (wt %) – C | Ash | Sorbent | Sorbent Composition (m %) CaO | $CaCO_3$ | $CaSO_4$ |
|---|---|---|---|---|---|---|---|---|---|---|
| 30.2 | Limestone | 2.5 | 670 | Bed-End of Run | Sample Not Taken | | | | | |
| | | | | Bed-Rejected Solid | 3.3 | 24 | 73 | 26 | 65 | 9 |
| | | | | Fly Ash | 5.0 | 78 | 16 | 38 | 12 | 50 |
| | | | | Fine Particulate | 4.1 | 79 | 16 | ~41 | 3 | 130 |
| 31.1 | Limestone | 3.0 | 840 | Bed-End of Run | 0.1 | 10 | 90 | 29 | 49 | 22 |
| | | | | Bed-Rejected Solid | 0.5 | 26 | 73 | 48 | 24 | 27 |
| | | | | Fly Ash | 10.1 | 65 | 26 | 43 | 13 | 44 |
| | | | | Fine Particulate | 7.2 | 80 | 13 | 11 | 14 | 76 |
| 32.3 | Dolomite | 1.5 | 840 | Bed-End of Run | 0.1 | 9 | 90 | 23 | 34 | 43 |
| | | | | Bed-Rejected Solid | Sample Not Taken | | | | | |
| | | | | Fly Ash | 12.2 | 58 | 30 | 42 | 12 | 46 |
| | | | | Fine Particulate | 2.4 | 71 | 26 | 64 | 4 | 32 |
| 36.2 | Dolomite | 1.3 | 900 | Bed-End of Run | N.D.(1) | 2(2) | 98(2) | 13 | 30 | 58 |
| | | | | Bed-Rejected Solid | Sample Not Taken | | | | | |
| | | | | Fly Ash | 2.0 | 41 | 56 | 38 | 7 | 55 |
| | | | | Fine Particulate | 3.6 | 73 | 23 | 30 | 6 | 64 |
| 37 | Dolomite | 4.5 | 900 | Bed-End of Run | N.D.(1) | 13(2) | 87(2) | 3 | 7 | 90 |
| | | | | Bed-Rejected Solid | Sample Not Taken | | | | | |
| | | | | Fly Ash | 2.0 | 48 | 49 | 14 | 5 | 81 |
| | | | | Fine Particulate | 3.6 | 79 | 17 | 35 | 2 | 63 |

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(1) N.D. – Not Determined

(2) Carbon Free Basis

concentrations of sorbent fines. The sorbent portion of the fine particulates generally ranges from 15 to 30% for dolomite sorbent and 10 to 20% for limestone sorbent. The ash content of the fine particulates generally ranges from 70 to 80% for both limestone and dolomite sorbent.

Combustion Efficiency

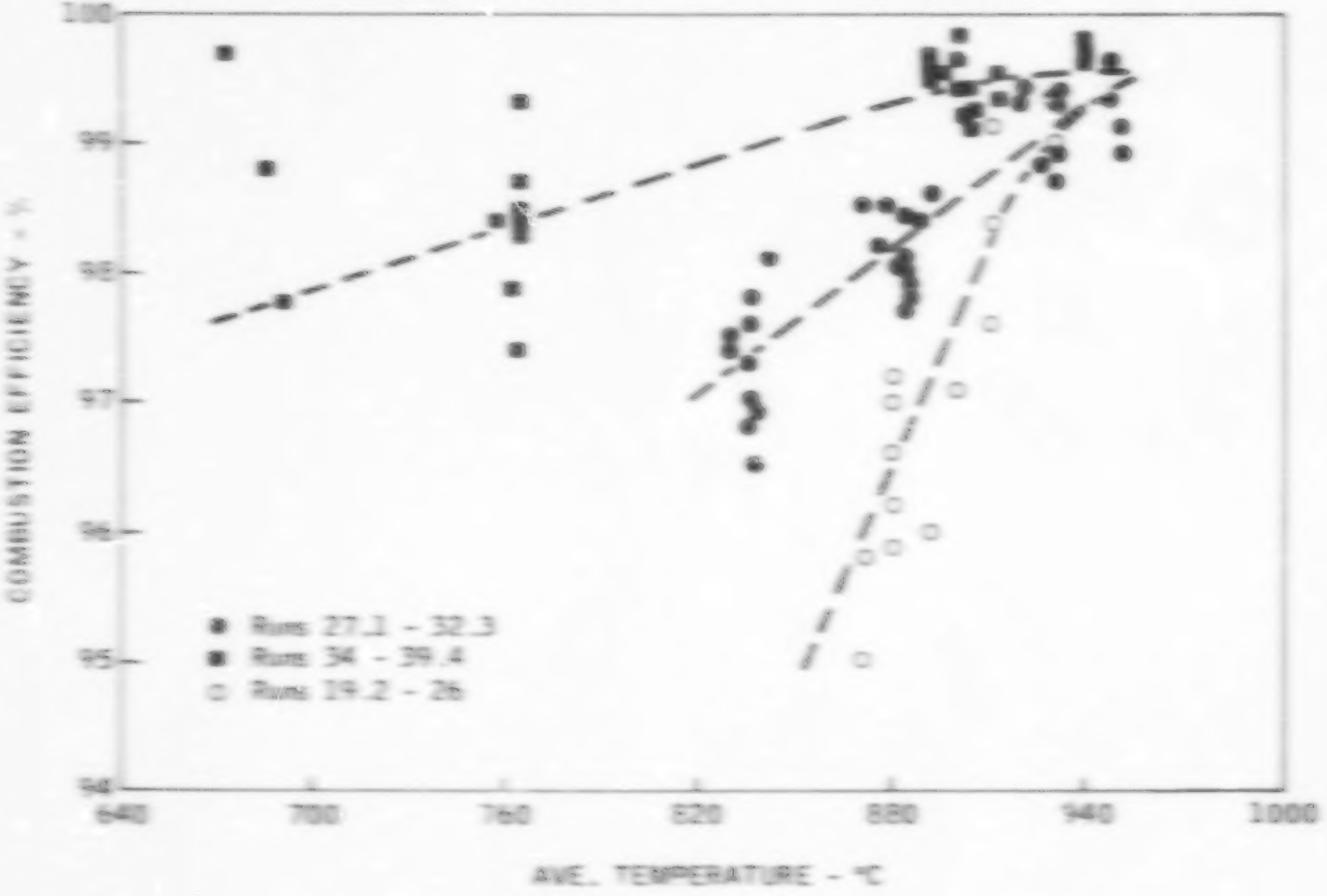
Combustion efficiency data are correlated as a function of the bed temperature in Figure III-29. The efficiency was calculated as one hundred minus the percentage of the combustible carbon in the feed lost with the flyash. Three distinct profiles were obtained depending on the run series. The profiles converge to 99 + 1% at 940°C. However, at lower temperatures, the temperature effect differs greatly. The lowest efficiencies were obtained from runs 19.2-26, and the highest efficiencies were obtained from runs 34-39.4. Results from runs 27.1-32.2 were between those from the other two series. The average efficiency at 880°C was, respectively, 96.5, 98.2 and 99.3%. The average efficiencies from the turndown tests were 98.2% at 760°C and 98.8% at 680°C. These values are much greater than the efficiencies which would be predicted from the two lower profiles. The data from runs 27.1-32.2 were examined for secondary variable effects using regression analysis. The excess air level, gas velocity, bed height and pressure were, however, not statistically significant.

The three temperature dependencies shown by the data in Figure III-29 have yet to be explained. A variety of explanations were considered, including process variables, the equipment, and sampling and analysis. The process variables examined were the coal feed rate, combustion intensity, excess air, velocity, sorbent, and coal chemical and size characteristics. None gave any indication of affecting the efficiency. Variations in the performance of the first and second stage cyclones and the first cyclone's flyash recycle system might affect the combustion efficiency. If the refractory lining was eroded or the barrel damaged, the collection efficiency would be impaired. This would mean a lower recycle rate from the first cyclone causing lower combustion efficiencies. The efficiencies were higher rather than lower in the later runs, however. The performance of the first cyclone could also be impaired by a backward gas flow in the cyclone's dipleg if a seal was not formed. Particle size analysis of the second stage cyclone collection and temperatures in the first cyclone dipleg during operation suggest that this did not happen. A poor second stage cyclone collection does cause carry over to the off gas of some carbon bearing flyash particles. This elutriated carbon is not included in the combustion efficiencies calculated for Figure III-29. The magnitude of this loss was evaluated from the chemical composition of the isokinetically sampled off gas particles and from a flyash balance. Table III-20 shows that carbon losses with the off gas particulates are small (0.2-0.4% of the feed carbon) and are independent of the flyash carbon content and cyclone collection efficiency. The different temperature dependencies in Figure III-29 are, therefore, not due to varying carbon losses in the off gas from the second cyclones.

The sampling procedure differed in Runs 19.2-26 from that used in later runs. In the earlier runs the carbon content of the entire flyash collection for a run was used in the calculation of the efficiency, including the non-representative amounts of unburned carbon remaining from startup and operating upsets. In the later runs, only the flyash collections made during a one to three hour period of stable operation were used for the carbon determination. There was an exception, Run 19.3, where three one-hour long collections were

FIGURE III-29

COMBUSTION EFFICIENCY VS. TEMPERATURE



made and analyzed for carbon. The combustion efficiency for these collections averaged 96.8% at 880°C as compared to 96.1% for several runs made at the same temperature but with the entire flyash collection used in the calculation. Apparently the difference in the sampling procedure cannot explain the difference in the profiles for Runs 19.2-26 and Runs 27.1-32.2. The sampling procedure was identical for Runs 27.1-32.2 and 34-39.4. The accuracy of the carbon determinations was confirmed with a known sample. Also, several flyash samples from early runs were reanalyzed with good agreement with the original results.

TABLE III-20. COMBUSTIBLE CARBON LOSSES

| Run No. | Second Cyclone Flyash Collection Eff. (%) | Combustible Carbon in the Second Cyclone Collection (% of Feed Carbon) | Combustible Carbon in Off Gas (% of Feed) |
|---|---|---|---|
| 31.1 | 83 | 1.1<br>1.5 | 0.2<br>0.2 |
| 32.2 | 53 | 0.7<br>0.6 | 0.6<br>0.6 |
| 32.3 | 55 | 3.0<br>2.2 | 3.2<br>0.2 |
| 36.2 | 71 | 0.5<br>0.7 | 0.2<br>0.4 |
| 37 | 60 | 0.6 | 0.2 |

## Heat Transfer Coefficients

Heat transfer coefficients were measured by maintaining the cooling water flow through the coils in the liquid state. The flow to each coil was measured with an orifice meter. The inlet and outlet temperatures of the cooling water were measured with thermocouples inserted within the piping. The flows and temperatures were recorded on the data logger at one minute intervals. These data were used to calculate the average coefficient and standard deviation for a 10 minute interval. Typical results from Run 19.2 are shown in Table III-21. The average of the three good measurements, for coils 1A, 1B, and 2B was 336 W/m² K which compares closely with a value of 358 W/m² K calculated from an overall heat balance. The cooling coils removed 57% of the heat input, and the remaining portion was removed as sensible heat by the fluidizing air or lost to the surroundings. The combustion intensity had a high value of about 5 MW/m³ expanded bed.

The heat transfer measurements made since run 19.2 are tabulated in Table III-22. Most of the data were obtained during the turndown tests. These data are the most consistent and were used to determine the effect of some of the operating parameters. The data are plotted in Figure III-30, against the bed temperature. The overall coefficient was lowered by 10% when the temperature was reduced from 950°C to 880°C. Some of the decrease may have been caused by a 20% reduction in superficial gas velocity at the lower temperatures. Much of the decrease probably occurred, however, because of a reduction in the radiation component of the heat flux. The magnitude of the

## TABLE III-21. MINIPLANT OVERALL HEAT TRANSFER COEFFICIENT MEASUREMENTS - RUN 19.2

| Coil # | Average Coeff. | Standard Deviations of 10 Measurements Obtained At One Minute Intervals |
|---|---|---|
| 1A | 352 W/m$^2$ K | 7.2 W/m$^2$ K |
| 1B | 318 | 7.7 |
| 2B | 329 | 11.3 |

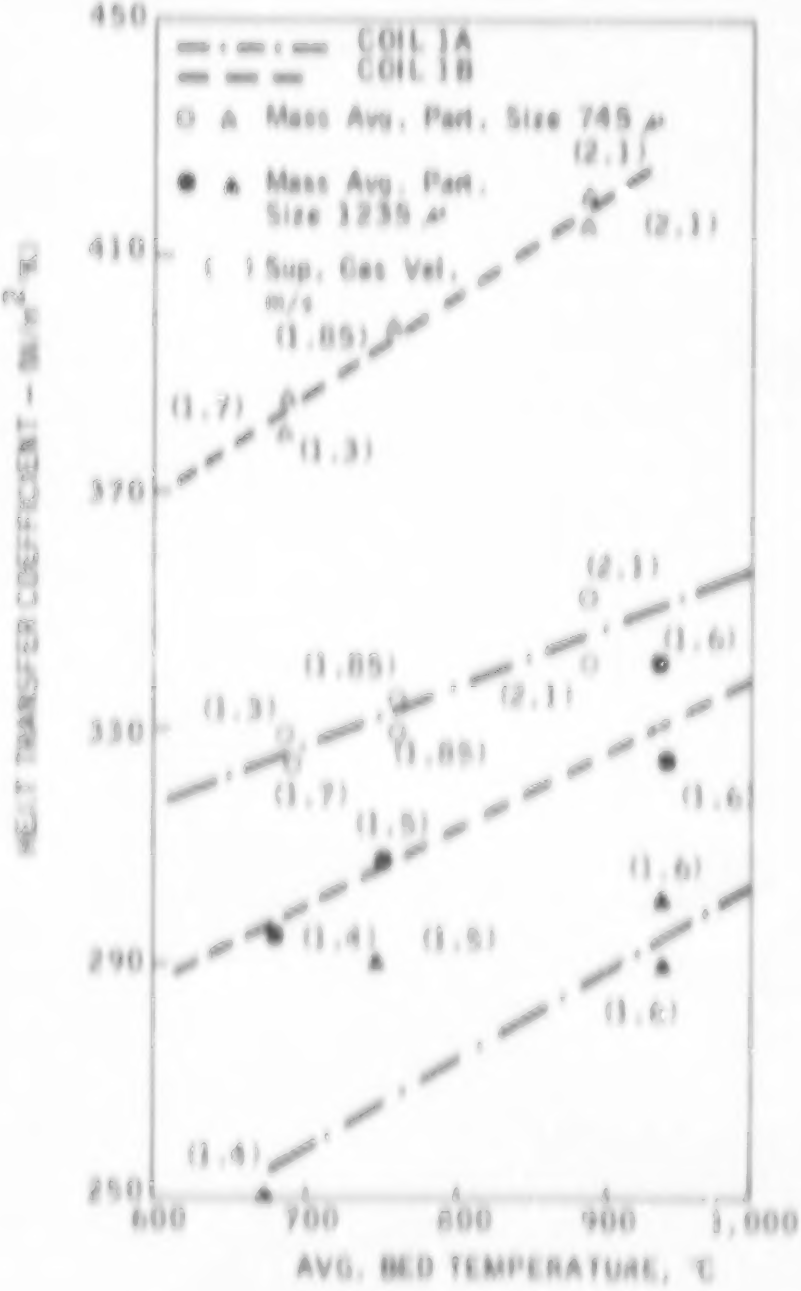
| | |
|---|---|
| Surface Area of a Coil, m$^2$ | 0.551 |
| Coil Heat Flux, W/m$^2$ | 280,000 |
| Combustion Intensity, W/m$^3$ Bed | 9,250,000 |
| Heat Removed by Cooling Coils in Bed, % of Coal Heat Input | 57 |
| Overall Heat Transfer Coeff., Calculated from Heat Balance, W/m$^2$K | 358 |

## TABLE III-22. HEAT TRANSFER COEFFICIENTS

| Run Number | Pressure (kPa) | Temp. °C | Sup. Vel. (m/s) | Mass Average Bed Part. Size (μ) | Overall Coefficient (W/m²K) | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | 1A | 1B | 2A | 2B |
| 19.2 | 930 | 880 | 1.9 | 1380 | 352 | 318 | 179 | 329 |
| 28.1 | 930 | 840 | 2.1 | 1089 | 352 | 346 | — | — |
| 29.1 | 930 | 875 | 2.2 | 1290 | 295 | 363 | 335 | 329 |
| 32.1 | 600 | 950 | 2.2 | 1060 | 273 | 341 | — | — |
| 38.1 | 930 | 890 | 2.1 | 745 | 341 | 415 | — | — |
| 38.2 | 930 | 890 | 2.1 | 745 | 352 | 420 | — | — |
| 38.3 | 930 | 760 | 1.85 | 745 | 329 | 398 | — | — |
| 38.4 | 930 | 760 | 1.85 | 745 | 335 | 398 | — | — |
| 38.5 | 930 | 685 | 1.7 | 745 | 324 | 386 | — | — |
| 38.6 | 930 | 685 | 1.3 | 745 | 329 | 380 | — | — |
| 39.1 | 930 | 750 | 1.5 | 1235 | 290 | 307 | — | — |
| 39.2 | 930 | 675 | 1.4 | 1235 | 250 | 295 | — | — |
| 39.3 | 930 | 940 | 1.6 | 1235 | 290 | 324 | — | — |
| 39.4 | 930 | 940 | 1.6 | 1235 | 301 | 341 | — | — |

FIGURE III-30

HEAT TRANSFER COEFFICIENTS VS. TEMPERATURE



radiation component was estimated by choosing, through trial and error, an emissivity which would make the outside film coefficient independent of the bed temperature. An emissivity of about 0.2 was calculated and the radiation component was estimated to be about 12% of the heat flux at 950°C.

The bed was much finer in runs with dolomite because dolomite has a higher attrition rate than limestone. The 40% difference in the mass average size, 763 μm vs. 1235 μm, in the turndown tests with dolomite and limestone increased the overall coefficient in the dolomite tests by 20 to 30%.

The location of the coil also had an effect on the coefficient. The lower most cooling coil 1A, located 0.46 m to 0.92 m above the fluidizing grid, had coefficients 15-20% lower than coil 1B, located 0.92 m to 1.38 m above the grid. The coefficient was largely independent of position among the coils located higher in the bed, when a number of coils greater than two was used. The difference between the coefficient for the lower two coils probably occurs because of varying mixing patterns. The gas jets from the fluidizing grid require a penetration depth before a uniform flow field is established and, therefore, the mixing may be more vigorous in the vicinity of the second coil.

After run 27 the vertical coils were baffled with rings to prevent erosion of the coils. The baffles which acted as fins added about 15% to the surface area of the coil. The average coefficient measured for the baffled coil compared closely with the average coefficient for unbaffled coils (331 vs. 335 W/m²K). It is possible that the stagnant areas created by the baffles reduce the bed to tube heat transfer coefficient slightly, thereby offsetting the increase in surface area.

## BATCH COMBUSTOR

### Equipment, Materials, Procedures

Fluidized Bed Coal Combustion Unit--
A schematic of the Exxon batch fluidized bed combustion unit is shown in Figure III-31. The fluidized bed combustor vessel was constructed from four sections of 25 cm diameter standard wall carbon steel pipe and refractory lined to an inside diameter of 11.6 cm. The height of the vessel above the fluidizing grid was about 6.9 m. Below the grid was a 61 cm long burner section lined with Greefco Bubblite. The fluidizing grid, which was made of stainless steel, had 21-0.32 cm diameter holes to distribute the fluidizing air. A natural gas burner, located below the grid, was used to preheat the unit to above the ignition temperature of coal. The combustor had three 0.95 cm diameter stainless steel vertical cooling coils extending from 27 to 144 cm above the grid. Each coil had a surface area of 0.060 m². Thermocouples were located 10 cm apart in the lower section of the combustor and 30 cm apart in the upper section.

Although sorbent material was added batchwise to the combustor, coal was fed continuously using a modified Petrocarb Model 10-1 ABC injector. The injector consists of a conical-bottom tank that holds coal to be fed and an orifice and mixing tee assembly that mixes coal with carrier gas. The coal feed rate is controlled by injector/combustor differential pressure and transport air flow rate.

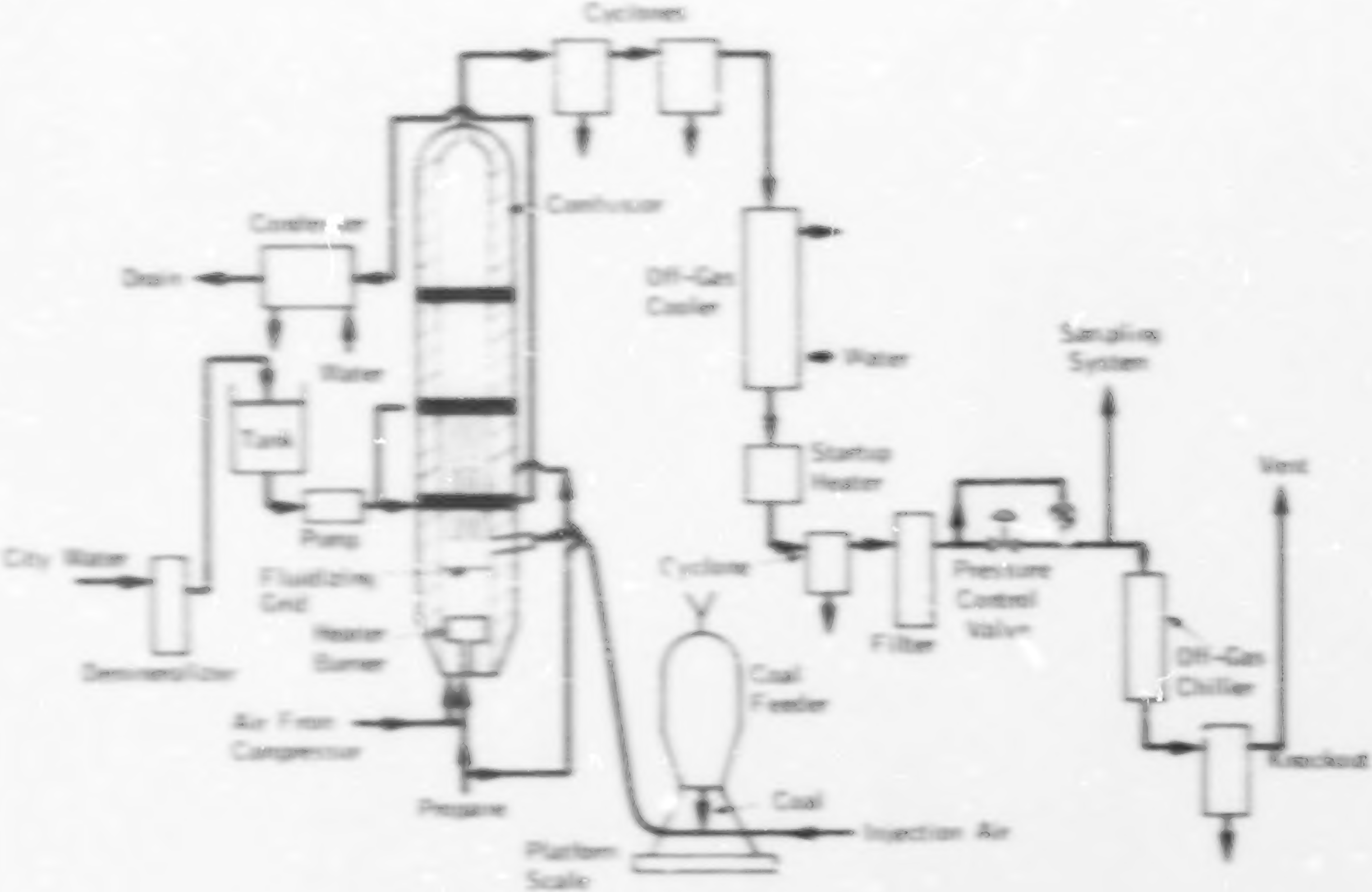

Cyclones
Combustor
Condenser
Drain
Water
Tank
Pump
City Water
Demineralizer
Fluidizing Grid
Heater Burner
Air From Compressor
Propane
Off-Gas Cooler
Water
Startup Heater
Cyclone
Coal Feeder
Coal
Injection Air
Platform Scale
Filter
Pressure Control Valve
Sampling System
Vent
Off-Gas Chiller
Knockout

FIGURE III-31

BATCH FLUIDIZED BED COAL COMBUSTION UNIT

Flow of air and fuel into the combustor and combustor pressure were under automatic control. Gases leaving the combustor first passed through two cyclones which removed flyash and entrained stone. An off-gas cooler, which followed the cyclones, reduced the temperature of the off-gas to the desired level. The off-gas then entered a 2.5 cm diameter stainless steel expansion coil which was electrically heated during startup to raise the temperature of the gas above the dew point. A 3.8 cm Aerotec cyclone, following the heater, was used to remove particulates during startup. Fine particulates were removed by a Pall Model MEC-800-18-C filter, located upstream of the back pressure control valve. Before being vented, the off-gas passed through a chiller and knockout for moisture removal. A small portion of the off-gas was extracted after the back pressure control valve and sent to the gas conditioning and analysis system for $SO_2$, $NO_x$, $CO_2$, and $O_2$ measurement.

A more detailed description of the components and development of the batch fluidized bed combustion unit can be found in an earlier report (2).

Coal/Sorbents Tested--

Three different coals were burned in the batch fluidized bed combustion unit. The majority of the runs were made using a high volatile (A) bituminous Eastern coal from the Arkwright mine ground to -16 mesh. Runs were also made using a low sulfur Western coal and a high sulfur Illinois coal. A proximate and ultimate analysis for each of the coals are presented in Table III-23.

Grove limestone and Tymochtee dolomite generally in the 8 x 25 mesh range were the primary sorbents used in the experimental studies. Baker dolomite and Pfizer dolomite were also tried but, because of high attrition rates, their use was discontinued. An analysis of the sorbents is given in Table III-24.

Experimental Procedures--

Operation of the batch fluidized bed combustor can be divided into four phases: startup, ignition and pre-heating, coal feeding, and shutdown. Startup consisted of those activities preliminary to ignition of the preheat burner. These activities included equipment checkout, calibration of the gas analyzers, charging solids, and turning on electrical circuits, the air compressor, the cooling water systems (fluidizing grid, burner, steam coils, condenser) and the purge air systems (pressure taps, sight-glasses, ΔP cells).

To ignite the preheat burner, air and fuel flows were set and the ignition electrode was activated. Safety devices shut down all flows if ignition was not obtained within ten seconds or if a flame-out occurred afterwards. A safety interlock prevented startup for 3 minutes after an automatic shutdown to assure adequate purging of the combustor. Subsequent to ignition, air flow and combustor pressure were adjusted to the desired values. All gas flows and pressure were controlled automatically. After the bed temperature was sufficiently high, supplementary fuel could be injected directly into the bed to reduce the time required to heat the bed to the coal ignition temperature.

Preparation of the coal feed system for a run consisted of setting the flow of injection air and activating and adjusting the coal feeder-to-combustor ΔP control system. Coal injection could be started only after the temperature in the combustor was high enough for self-ignition of the coal

### TABLE III-23. COMPOSITION OF COALS USED IN BATCH FLUIDIZED BED COAL COMBUSTION PROGRAM

|  | Proximate Analysis |  |  |  | Ultimate Analysis |  |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Coal | Moisture | Ash | Volatiles | Fixed Carbon | Moisture | Ash | Total Carbon | Hydrogen | Sulfur | Nitrogen | Chlorine | Oxygen(1) |
| Arkwright | 1.00 | 8.11 | 36.86 | 54.03 | 1.00 | 8.11 | 76.26 | 5.30 | 2.66 | 1.49 | 0.07 | 5.11 |
| Illinois | 3.67 | 10.25 | 39.50 | 46.59 | 3.67 | 10.25 | 67.23 | 4.79 | 4.24 | 1.19 | 0.07 | 8.56 |
| Western | 19.45 | 7.08 | 36.89 | 36.58 | 19.45 | 7.08 | 55.12 | 3.56 | 0.60 | 0.77 | 0.02 | 13.02 |

Notes: All values are weight percent

(1) by difference

TABLE III-24. PROPERTIES OF LIMESTONE AND DOLOMITE

| Designation | Quarry Source | Stone Type | Chemical Analysis, Wt. % | | | | |
|---|---|---|---|---|---|---|---|
| | | | CaO | MgO | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ |
| 1359 | Grove Limestone<br>(Stephens City, Va) | Limestone | 97.0 | 1.2 | 1.1 | 0.3 | 0.2 |
| 1337 | Chas. Pfizer Co.<br>(Gibsonburg, Oh) | Dolomite | 54.0 | 44.0 | 0.9 | 0.2 | 0.3 |
| Tymochtee | C. F. Duff & Sons<br>(Huntsville, Oh) | Dolomite | 53.8 | 38.7 | 5.3 | 0.6 | 1.2 |

to occur. Flow of the preheat fuel was stopped automatically at the same time that feeding of coal was started. An automatic safety circuit would shut down coal injection if the combustor temperature dropped too low to ensure combustion of the coal or if the feeder-to-combustor ΔP dropped below a pre-set minimum (about 6.9 kPa).

The weight of the coal feeder vs. time was taken so that the feed rate of coal could be determined. Another method of estimating the feed rate was to observe the oxygen concentration in the off-gas from the combustor. A rapid rise in oxygen concentration was usually the quickest way of determining that a problem was developing with the coal feeding system. Combustor temperature could be controlled by regulating the amount of coal burned. The feed rate of coal could be adjusted by changing the flow of injection air or coal feeder-to-combustor ΔP.

To shut down the combustor routinely, the coal feed valve was closed, fluidizing air was stopped, and nitrogen purge was started to preserve the solids. Flow of injection air was kept on for several minutes so that the coal feed line could be cleared of coal. All water flows were gradually reduced. Solids could be discharged from the reactor by blowing them out of a port located just above the fluidizing grid after the combustor had cooled.

## Batch Combustor Performance

A detailed discussion of the development of the primary components of the batch combustor was presented in a previously issued report (2). Included were discussions of the development of the modified Petrocarb coal feed system, cooling coil design, and sampling system. These components continued to perform satisfactorily and no additional modifications were required. Additional modifications were made to other parts of the unit to improve performance. These are described below.

### Bed Preheat System--

The preheat system was modified so that propane could be injected directly into the bed at a position approximately 15 cm above the fluidizing grid to supplement the main propane supply during preheat. The auxiliary propane is injected using a "sonic" air jet type nozzle to promote better mixing of the propane in the bed. The use of the air jet nozzle was found necessary to prevent the propane from burning above the bed. The procedure for preheat consisted of using the main propane supply to bring the bed temperature to approximately 500°C whereupon the auxiliary fuel was injected. When the temperature started to rise rapidly, the main fuel supply was decreased to provide sufficient air for combustion of the auxiliary propane. This new preheat technique allowed the bed to be preheated with flow to all three cooling coils. This prevented the coils from experiencing high metal temperatures and was intended to help prevent possible high temperature corrosion. This preheat technique also placed less stress on the fluidizing grid.

The preheat system was later modified to use natural gas rather than propane.

### Bed Agglomeration - Grid Design--

A problem which occurred periodically during batch combustor operation was that of bed agglomeration. Bed agglomeration normally occurs when the ash in the coal begins to molten and stone particles begin to adhere. Conditions during which agglomeration usually occurred resulted from either poor solids mixing and heat removal or sudden surges of coal into the combustor. To promote better solids mixing a new fluidizing grid was designed. The new grid had 21-0.3 cm diameter air distribution holes as compared to 80-0.16 cm diameter holes in the old grid. After the installation of the new grid, bed agglomeration was not a problem.

In order to provide better coal feed control, the location of the pressure tap for the low pressure side of the coal feed ΔP controller was moved from a position within the bed to the freeboard region. The purpose of this change was to insure that this tap would not plug and thereby interfere with the ΔP control.

## Batch Combustor Results

During the course of the batch unit variables study, the following coal/sorbent combinations were tested:

- Eastern (Arkwright) coal/Grove limestone
- Eastern (Arkwright) coal/Tymochtee dolomite
- Illinois #6 coal/Grove limestone
- Western coal/Grove limestone

A summary of the run conditions is given in Appendix Table M. Results are given in Appendix Tables N and O.

### $SO_2$ Emissions--

$SO_2$ emissions measured during batch unit runs varied with time, increasing as the bed became more sulfated. Runs were made to various $SO_2$ concentrations in the flue gas, the bed analyzed for $SO_4^=$, and the equivalent calcium to sulfur molar ratio calculated as

$$Ca/S = \text{fraction } SO_2 \text{ removed/fraction of calcium sulfated}$$

The emission at the end of the run was then plotted against the equivalent calcium to sulfur ratio. It had been planned to calculate equivalent calcium to sulfur ratios for periods before the end of a run by calculating a sulfur mass balance and using the final sulfation level as an anchor point. However, this approach was not workable since a portion of the bed was lost by attrition and entrainment during the run and corrections for bed loss could not be made accurately.

$SO_2$ emissions are given in Appendix Table N. They are shown as a function of the equivalent calcium to sulfur ratio for runs burning Arkwright (Eastern) coal and using limestone and dolomite as the $SO_2$ sorbent in Figures III-32 and III-33. Figures III-34 and III-35 show the same data expressed as percent $SO_2$ retention vs. the calcium to sulfur ratio. Data were obtained at a pressure of 800 kPa, with the exception of two runs made at 310 and 395 kPa, vs. the calcium to sulfur ratio. Data were obtained at a pressure of 800 kPa, at superficial velocities of 0.9 to 1.9 m/s, over a temperature range of 750-975°C. As shown in the figures, the data

FIGURE III-32

$SO_2$ EMISSIONS VS Ca/S RATIO - EASTERN COAL AND LIMESTONE No. 1359

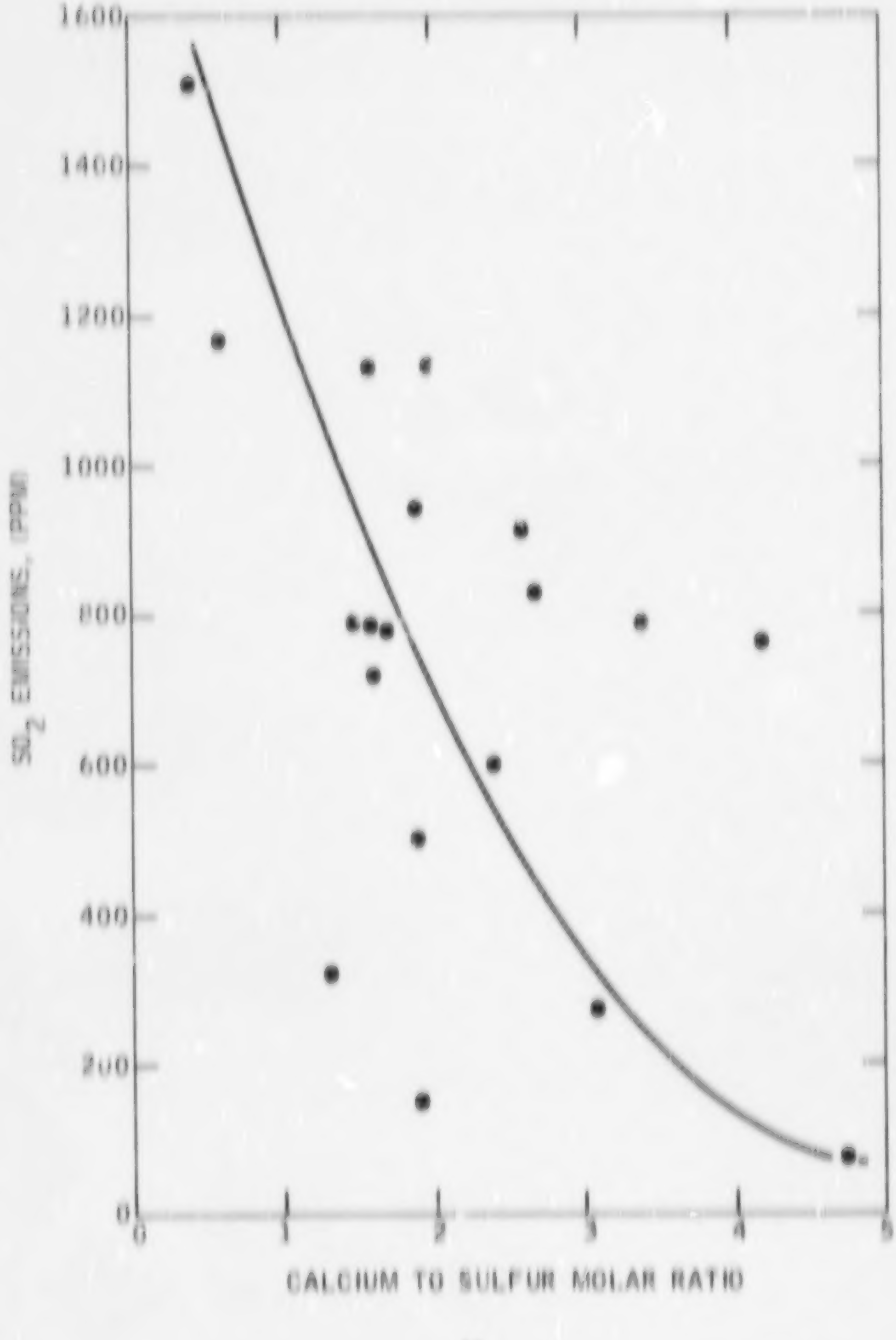


FIGURE III-33

$SO_2$ EMISSIONS VS Ca/S RATIO-EASTERN COAL AND TYMOCHTEE DOLOMITE

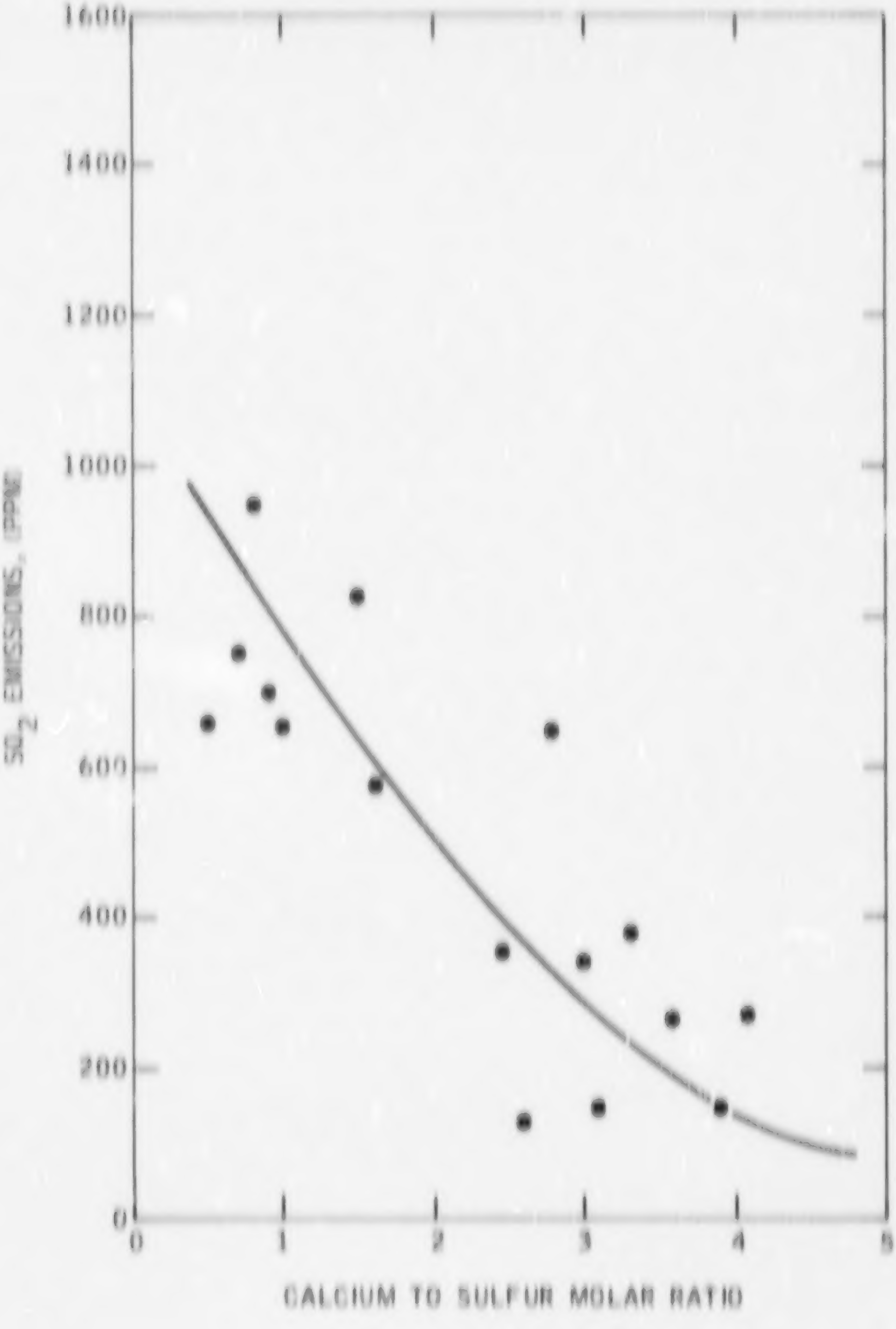


FIGURE III-34

$SO_2$ RETENTION VS Ca/S - EASTERN COAL -
LIMESTONE No. 1359

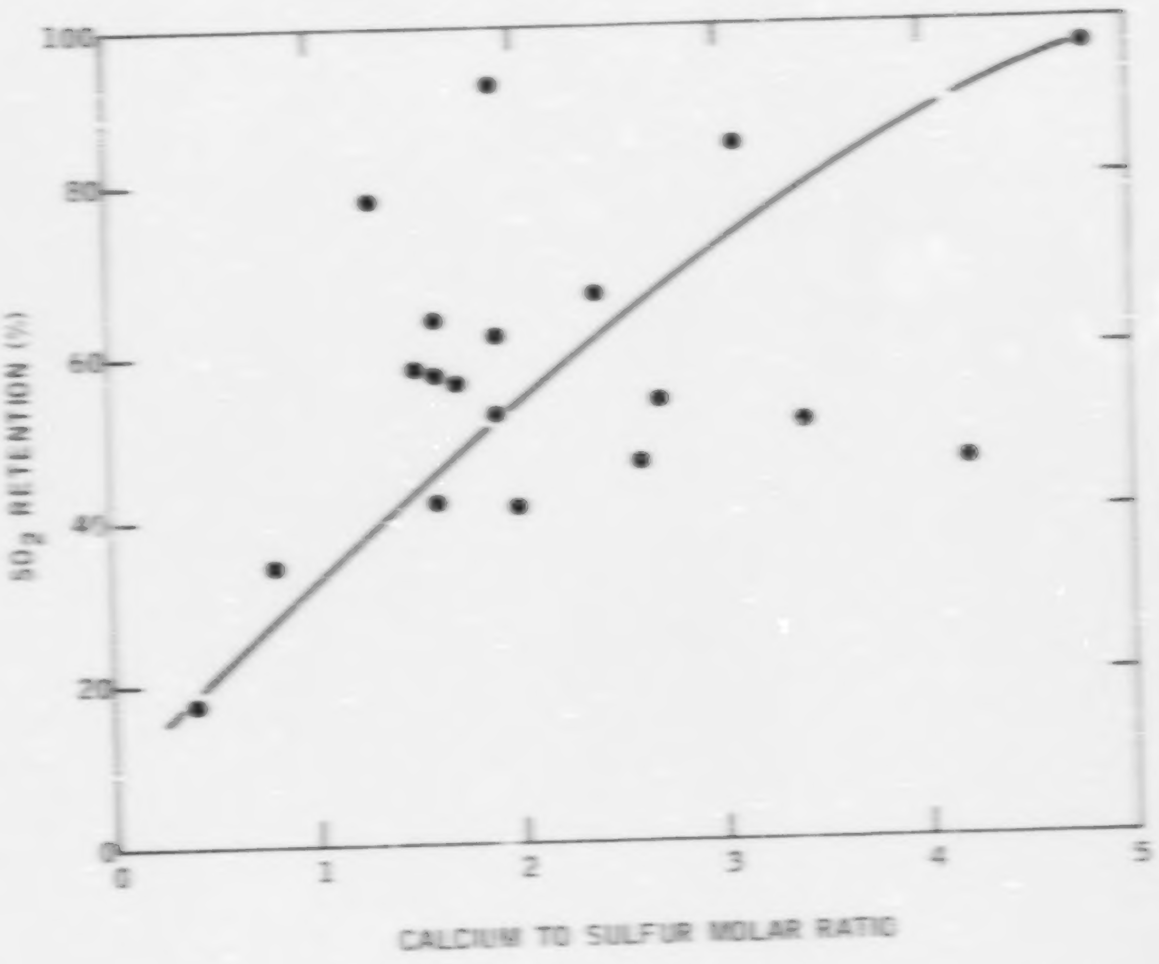
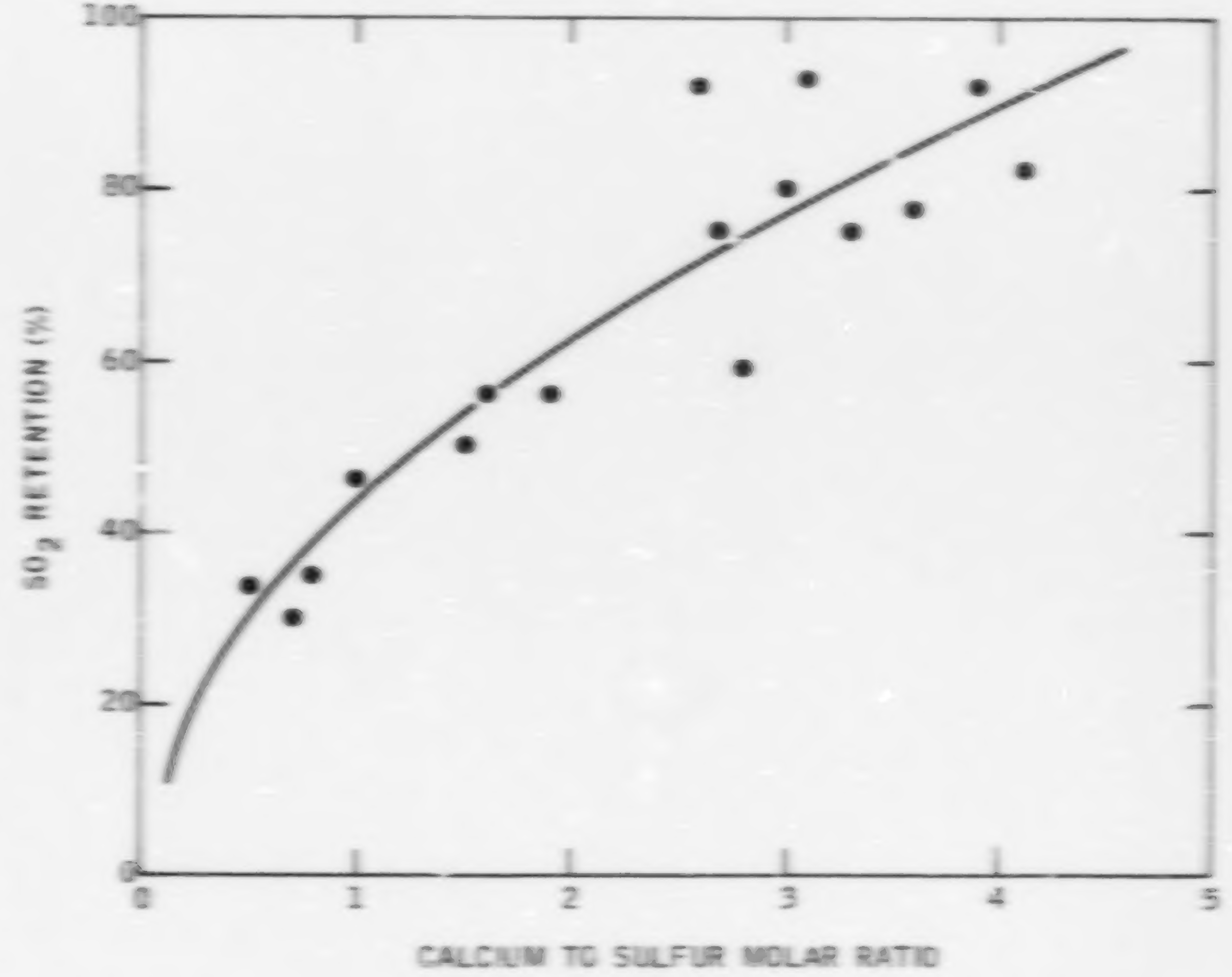


FIGURE III-35

$SO_2$ RETENTION VS Ca/S RATIO - EASTERN COAL - TYMOCHTEE DOLOMITE



obtained exhibited considerable scatter. As a result of this data scatter, difficulty in assessing the effect of temperature and excess air was encountered. Part of the scatter can probably be attributed to the fact that the batch unit does not operate in a steady state fashion. Operation of the unit is characterized by constantly changing $SO_2$ emissions and bed sulfation levels, as well as decreasing bed depths and particle sizes due to attrition and entrainment.

Two other factors which have been shown to affect $SO_2$ retention with limestone sorbent are the differences in the extent of calcination and gas phase residence time. This was noted in previous sections describing miniplant $SO_2$ emissions results (p. 36 and 40). A significant improvement in the $SO_2$ vs Ca/S correlation occurred when these factors were taken into account. The effect of variable calcination levels was accounted for by using an empirically modified form of the Ca/S ratio which considers differences in the degree of calcination. To do this, an effective Ca/S ratio was calculated as follows:

$$\text{Effect. Ca/S Ratio} = (\text{Ca/S}) \cdot X_{(CaO\ +\ CaSO_4)}$$

where $X_{(CaO\ +\ CaSO_4)}$ is the mole fraction of the calcium present as the oxide or sulfate.

This approach is similar to that applied to the miniplant data (p. 52). Use of this effective Ca/S ratio decreased the extent of data scatter as shown in Figure III-36. However, this approach is presented only as a means of explaining the relatively poor reproducibility when limestone sorbent is used. It cannot be used to predict desulfurization results with limestone sorbent, since the degree of calcination cannot as yet be predicted. Predictions of desulfurization results with limestone sorbent must be based on correlations such as those given in Figures III-18 or III-34, which are not corrected for variable calcination levels.

Correcting for variations in residence time has also produced less data scatter in batch unit data. The corrections were based on the following assumptions:

- a first order sulfation reaction
- complete mixing of bed solids
- plug flow of combustion gases through the combustor

Figure III-37 shows the limestone retention results corrected to the average retention time of 0.64 seconds while the dolomite $SO_2$ retention results for an average retention time of 0.76 seconds are shown in Figure III-38. As seen, the extent of data scatter was reduced for limestone. A comparison of Figures III-37 and III-38 also indicates that, as had been reported previously (p. 37), dolomite is a more effective sorbent than limestone at an equivalent Ca/S ratio.

Data obtained from the Illinois #6 coal/limestone test series are presented in Figure III-39. The effect of temperature is more visible during these runs than during the earlier limestone runs burning Arkwright (Eastern) coal. However, for reasons still unknown, $SO_2$ retention was lower than anticipated based on data obtained during the Arkwright coal/limestone test series.

FIGURE III-36

$SO_2$ RETENTION VS EFFECTIVE Ca/S RATIO -
LIMESTONE No. 1359

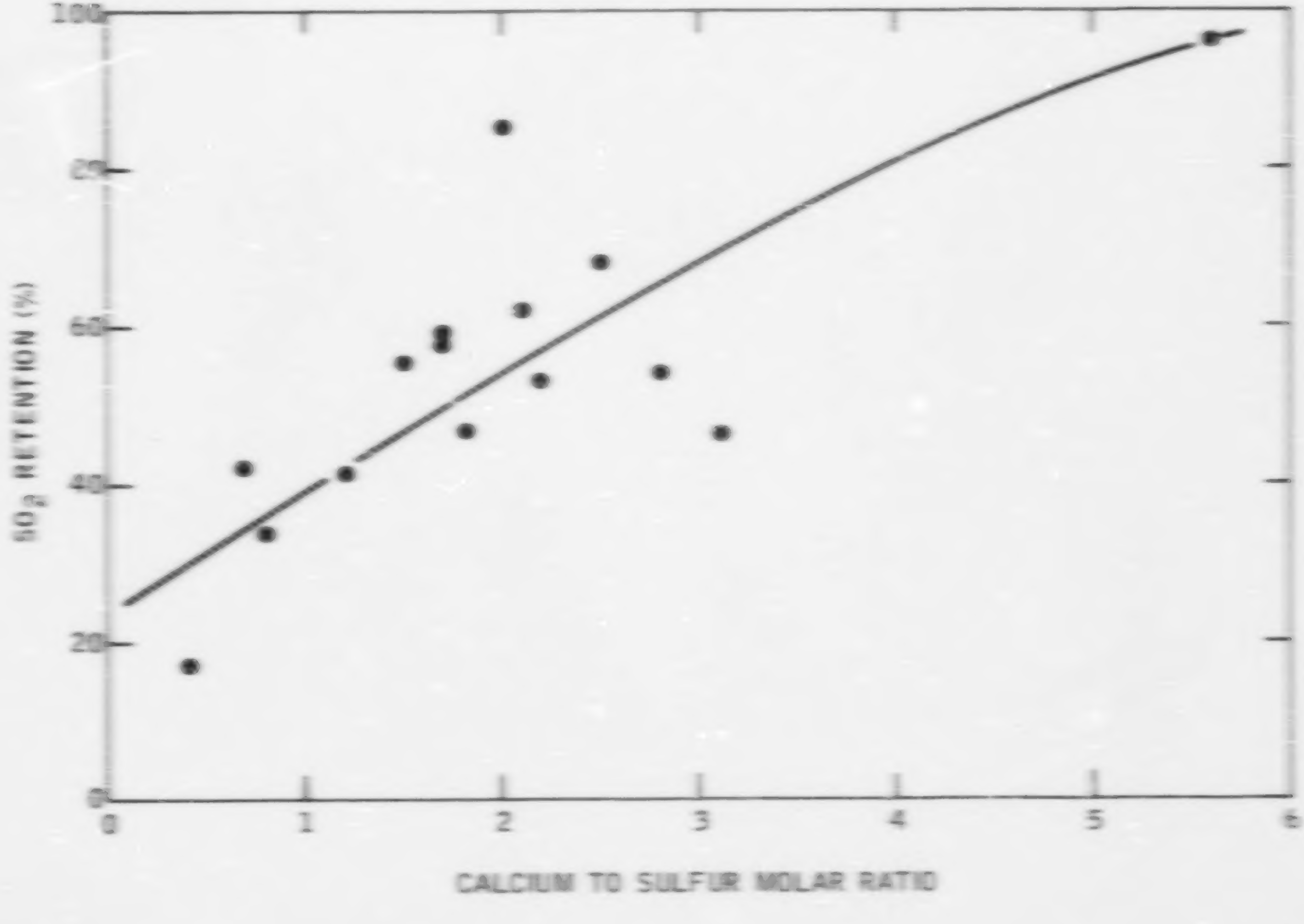


FIGURE III-37

$SO_2$ RETENTION AT CONSTANT RESIDENCE TIME
LIMESTONE No. 1359

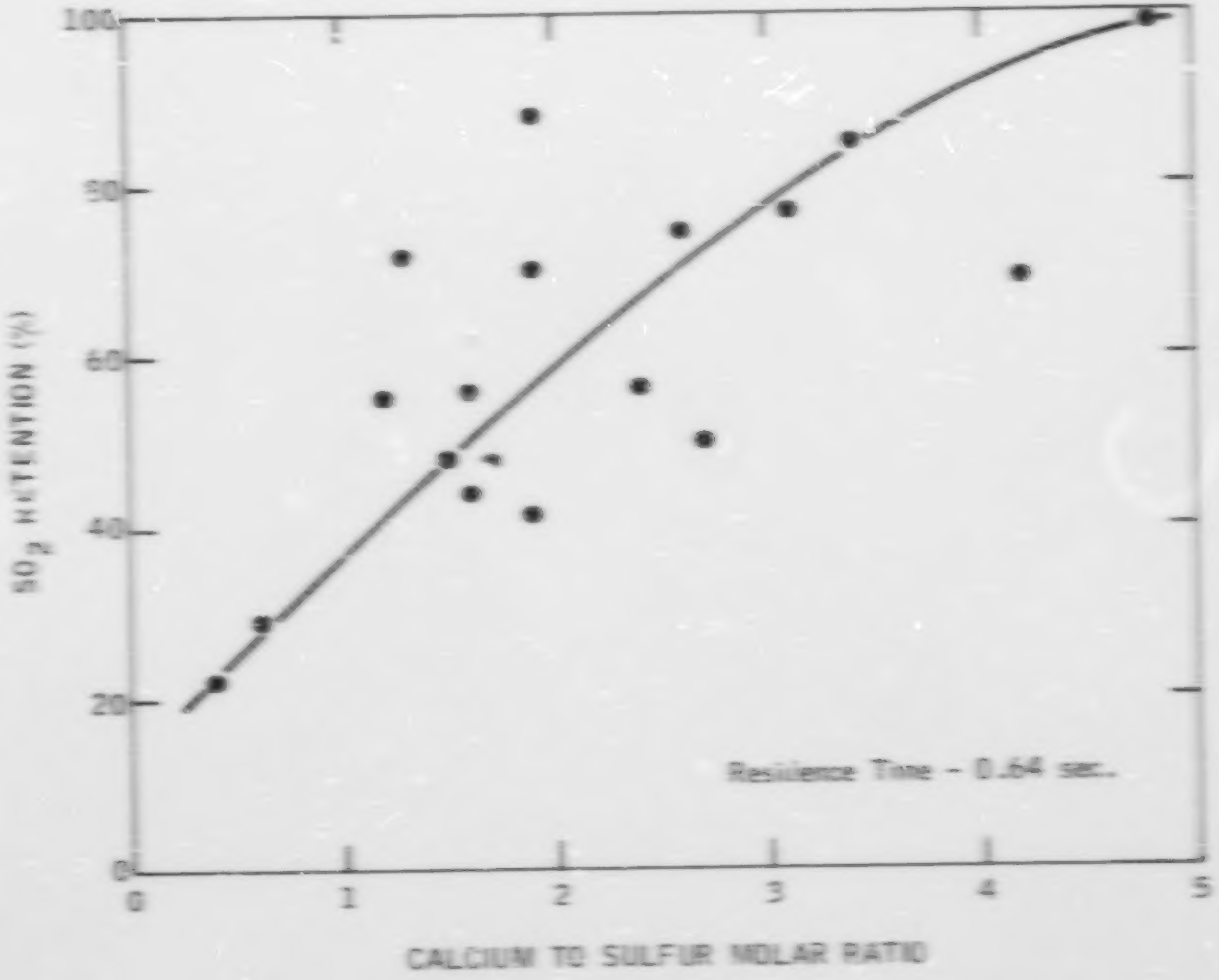


FIGURE III-38

$SO_2$ RETENTION AT CONSTANT RESIDENCE TIME
TYMOCHTEE DOLOMITE

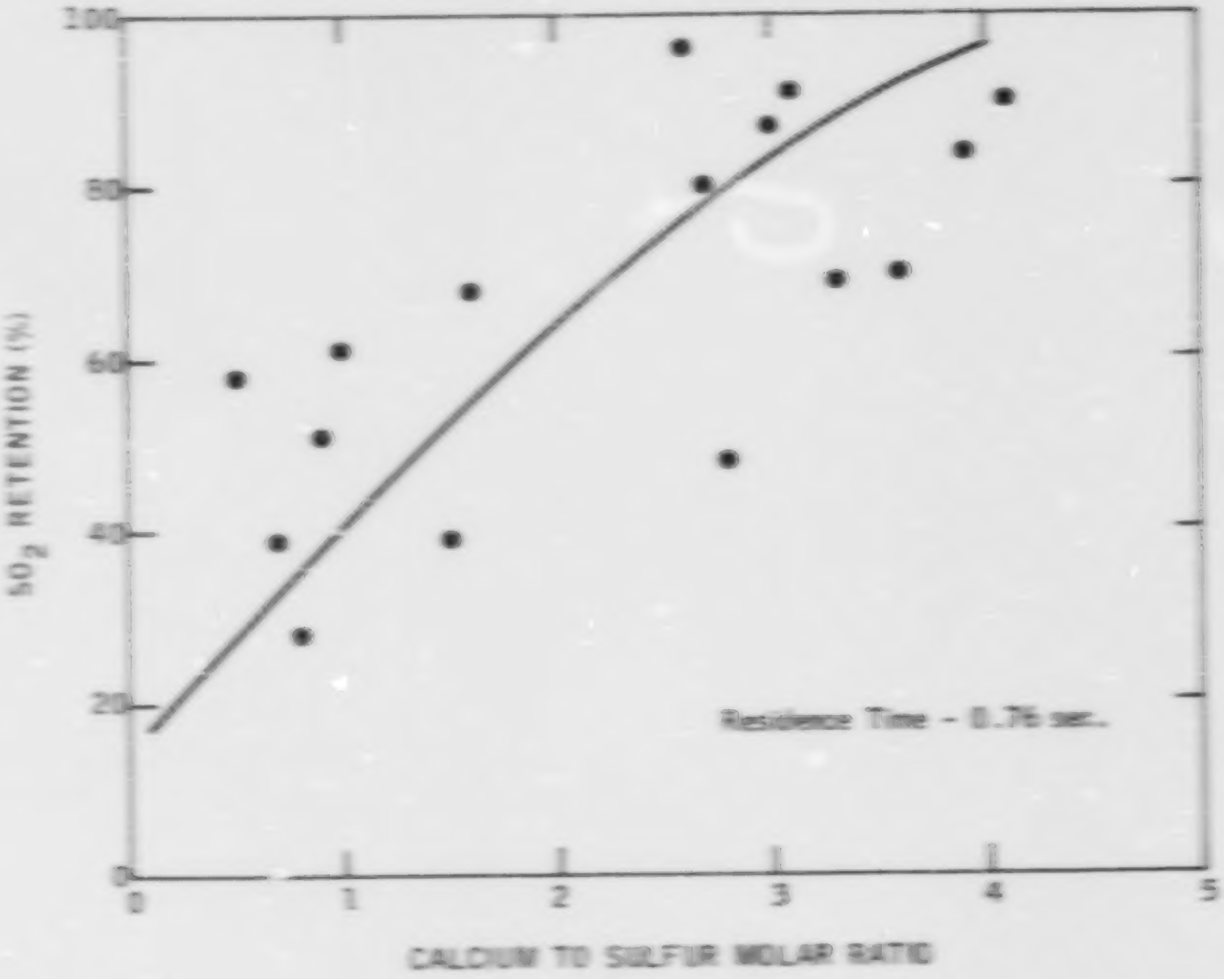
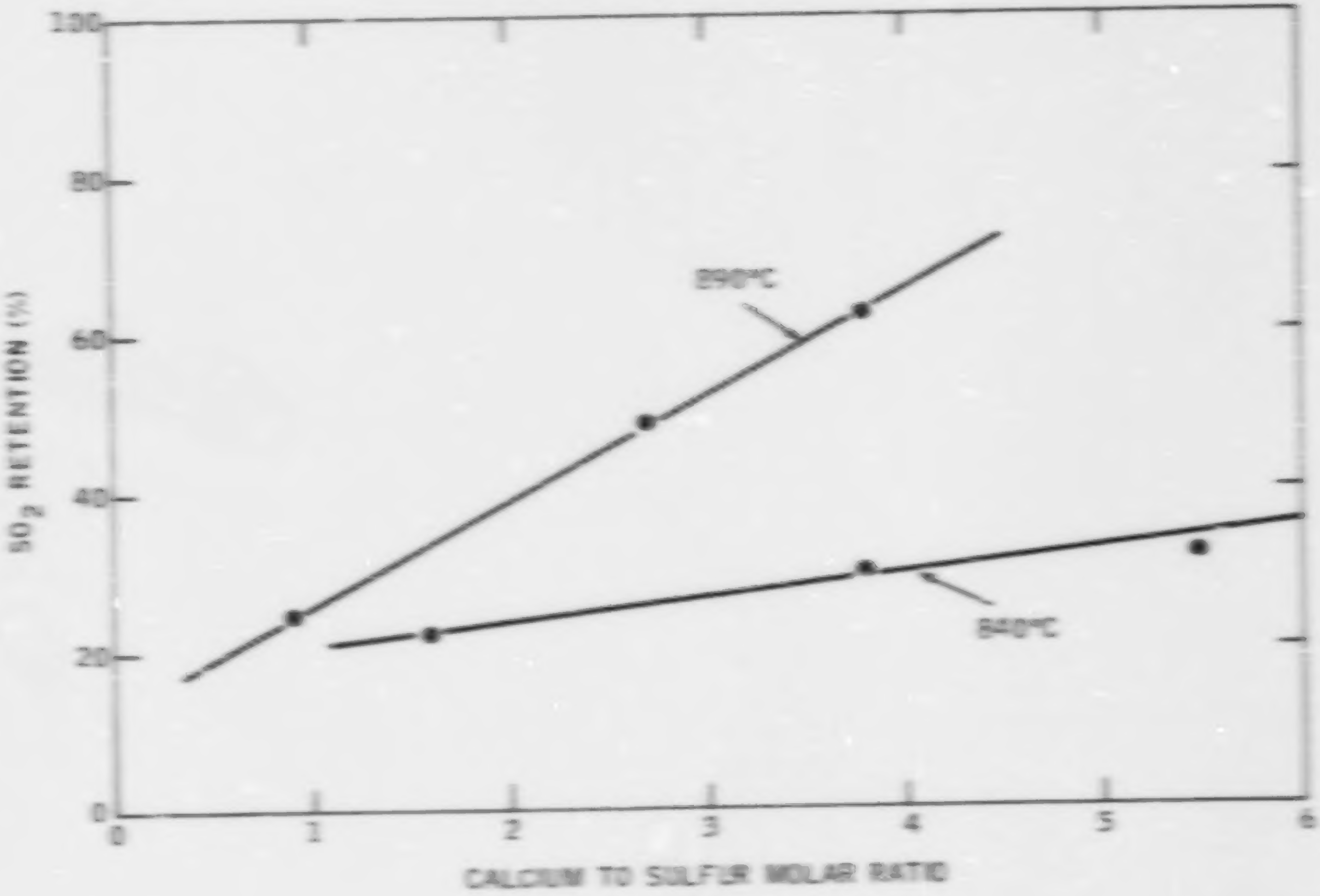


FIGURE III-39

$SO_2$ RETENTION VS Ca/S RATIO - ILLINOIS No. 6 COAL - LIMESTONE No. 1359



A total of 5 runs were also made in the batch unit burning a low sulfur (0.6%) Western coal and using Grove limestone as the $SO_2$ sorbent. The same bed material was used in each of the runs and a total of 105 kg of coal was burned. Although all of the runs were made at or below the limestone calcination temperature, the measured $SO_2$ levels were nevertheless extremely low. A possible explanation is that the $SO_2$ might have been reacting with the CaO contained in the ash. The calcium content of the coal was calculated to be equivalent to a Ca/S ratio of 1.5. To determine a baseline $SO_2$ emission level for the Western coal, a run was made using an inert bed. An $SO_2$ reduction of ~50% was observed during this run which further indicates that sulfur might be retained by the ash. The $SO_2$ level measured during this run is consistent with those reported by others (9) when burning a high calcium content lignite. These results are shown in Table III-25.

TABLE III-25. DESULFURIZATION OF WESTERN COAL

| Run No. | Sorbent | Temp. (°C) | % $SO_2$ Retention |
|---|---|---|---|
| 102 | Limestone | 840 | 100 |
| 103 | Limestone | 825 | 100 |
| 105 | Limestone | 850 | 88 |
| 106 | Limestone | 850 | 93 |
| 107 | Limestone | 845 | 94 |
| 104 | Alundum | 835 | 46 |

$SO_3$ Emissions--

$SO_3$ levels were determined during several runs using the method described previously on p. 61. Samples for the wet chemical analysis were extracted from the ducting just downstream of the combustor pressure control valve. The measured $SO_3$ levels were higher than expected and additional measurements must be made before any conclusions can be drawn.

$NO_x$ Emissions--

A regression analysis of $NO_x$ emission data was made and the results indicate that most of the variation could be explained by changes in the percent excess air. Figure III-40 shows $NO_x$ emission measured during batch unit runs (for all coals except the Western coal) plotted as a function of percent excess air. Although there is considerable scatter, the $NO_x$ emissions seem to tend to level out as percent excess air increases. However, when data from runs using Western coal are included, the data seem to fit a straight line as seen in Figure III-41. It is not certain whether the lack of curvature is really due to a difference in the emissions from the Western coal or if the line should be straight in Figure III-40. Unfortunately no data were obtained at the higher excess air levels for coals other than Western.

Data from all runs are also given in Appendix Table H. The data were obtained at a pressure of 800 kPa and at temperatures generally in the range of 800 to 975°C.

## FIGURE III-40

## BATCH UNIT $NO_x$ EMISSIONS

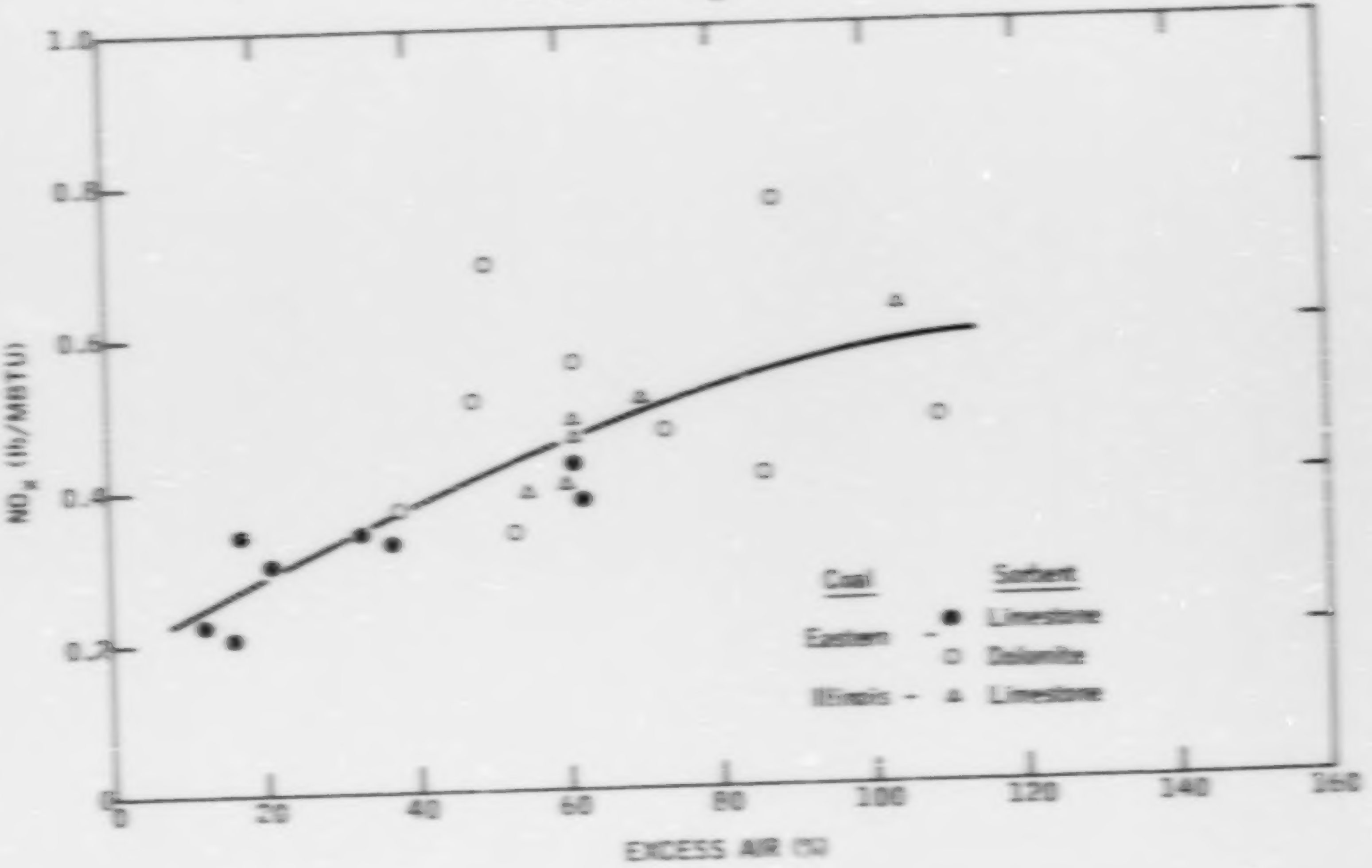
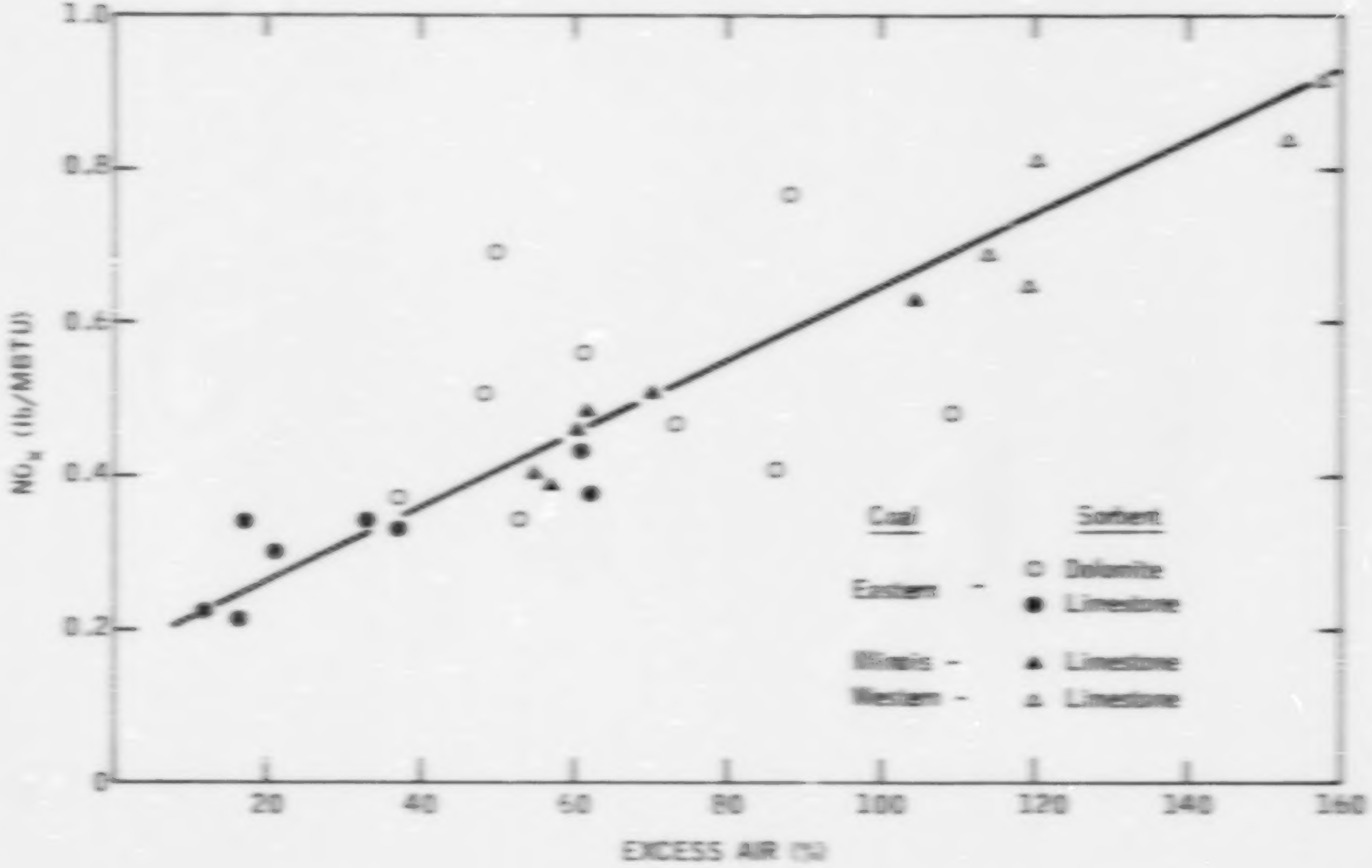


FIGURE IV-41

BATCH UNIT $NO_x$ EMISSIONS INCLUDING WESTERN COAL RESULTS



One important observation noted was that as the $SO_2$ concentration in the flue gas increased as a run progressed, the $NO_x$ emissions decreased. This decrease in the $NO_x$ emission may have occurred because of a reaction between $SO_2$ and NO. This fact, coupled with high excess air levels, may explain why the $NO_x$ emissions were very high in the runs burning low sulfur Western coal since very low $SO_2$ emissions were measured in these runs.

At excess air levels of 15-20%, the range anticipated for commercial FBC units, the emissions were generally in the range of 0.2 to 0.3 lb $NO_2$/M BTU. These levels are well below the EPA emission standard of 0.7 lb $NO_2$/M BTU. However, as seen in Figures III-40 and III-41, $NO_x$ levels were much higher at higher excess air levels, reaching 0.6 lb/M BTU at 100% excess air, and 0.9 lb/M BTU at 150% excess air with Western coal.

The $NO_x$ produced by the batch unit combustor was predominantly NO. Less than 5% was present as $NO_2$ and this was probably formed in the sampling system, since the equilibrium concentration of $NO_2$ is very low at the high temperatures occurring in the combustor.

CO Emissions--

The batch combustor CO level has been found to be dependent on the excess air level, the average bed temperature, and the steadiness of the coal feed rate. The excess air level appears to be a significant variable, especially at lower levels. Temperature appears to become significant only at very low levels (800°C). Prior to modifications in the coal feed system, CO emissions were generally quite high. The high levels were attributed to the unsteady coal feed rate which resulted in poor coal combustion. CO emissions averaged 960 ppm during these runs. Modifications to the coal feed system were made after Run 46C and after that the CO emissions averaged 180 ppm for temperatures above 800°C. At 750°C, a CO emission of 3600 ppm was measured. The effect of the steadiness of the coal feed rate will be discussed in more detail in the following section. CO data are given in Appendix Table O.

Combustion Efficiency--

Batch unit combustion efficiencies normally varied from 87 to 99% and were generally in the mid-90's. Combustion efficiency as a function of excess air and temperature is shown in Figure III-42. Data are also given in Appendix Table N. In one run in which both temperature and excess air were extremely low (620°C and 0%, respectively), a low combustion efficiency of 60% was calculated.

Another parameter which affected combustion efficiency was the steadiness of coal feeding. When coal feeding was unsteady, combustion efficiencies were consistently lower. Figure III-43 shows the combustion efficiency as a function of excess air for runs made both before and after modifications were made to the coal feed system. These modifications which involved the redesign of the orifice assembly on the coal feed vessel resulted in a significant improvement in the combustion efficiency. Over the entire range of excess air levels, the combustion efficiency increased between 3 and 4%. Batch unit combustion efficiencies are now more consistent with those reported from miniplant runs. A more detailed comparison of results from the two units is made in Section V.

FIGURE III-42

COMBUSTION EFFICIENCY VS EXCESS AIR AND TEMPERATURE

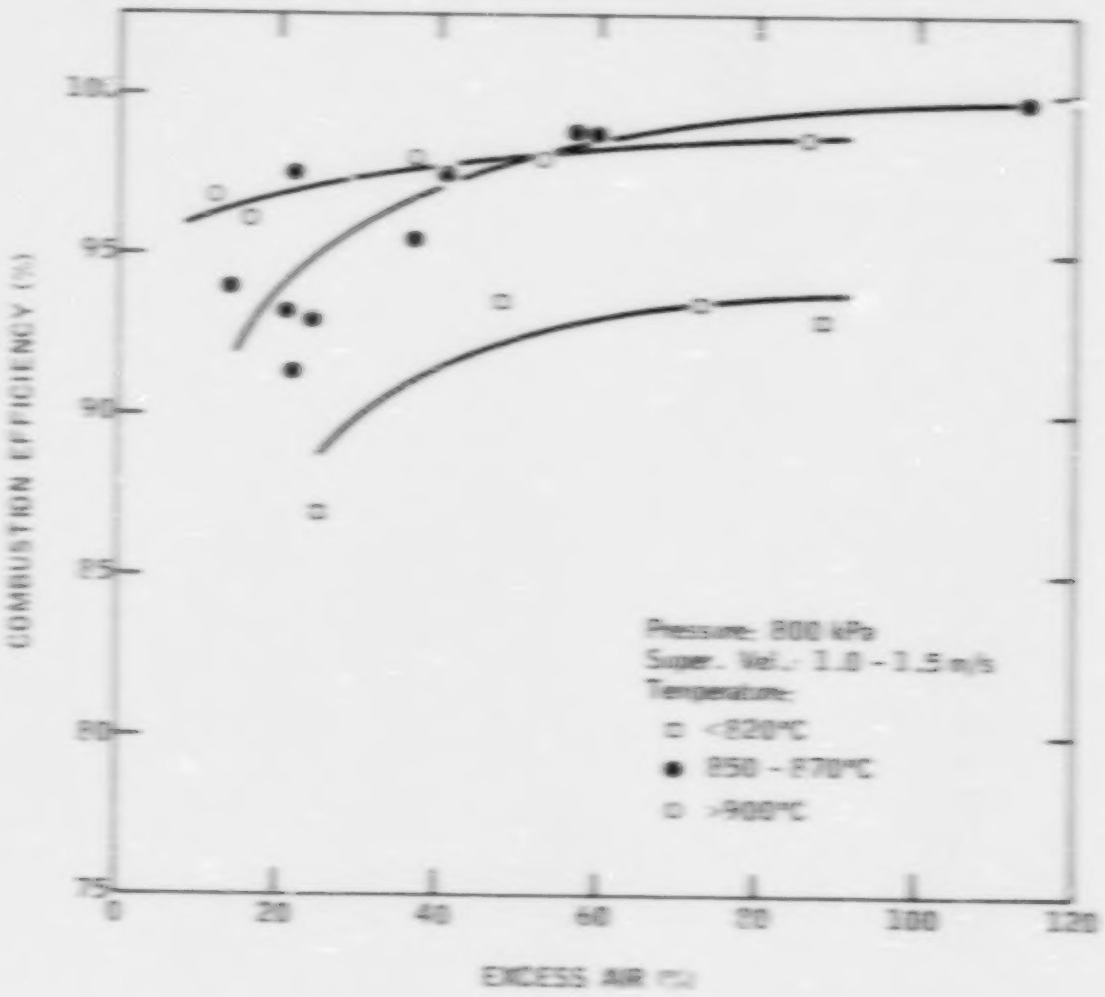
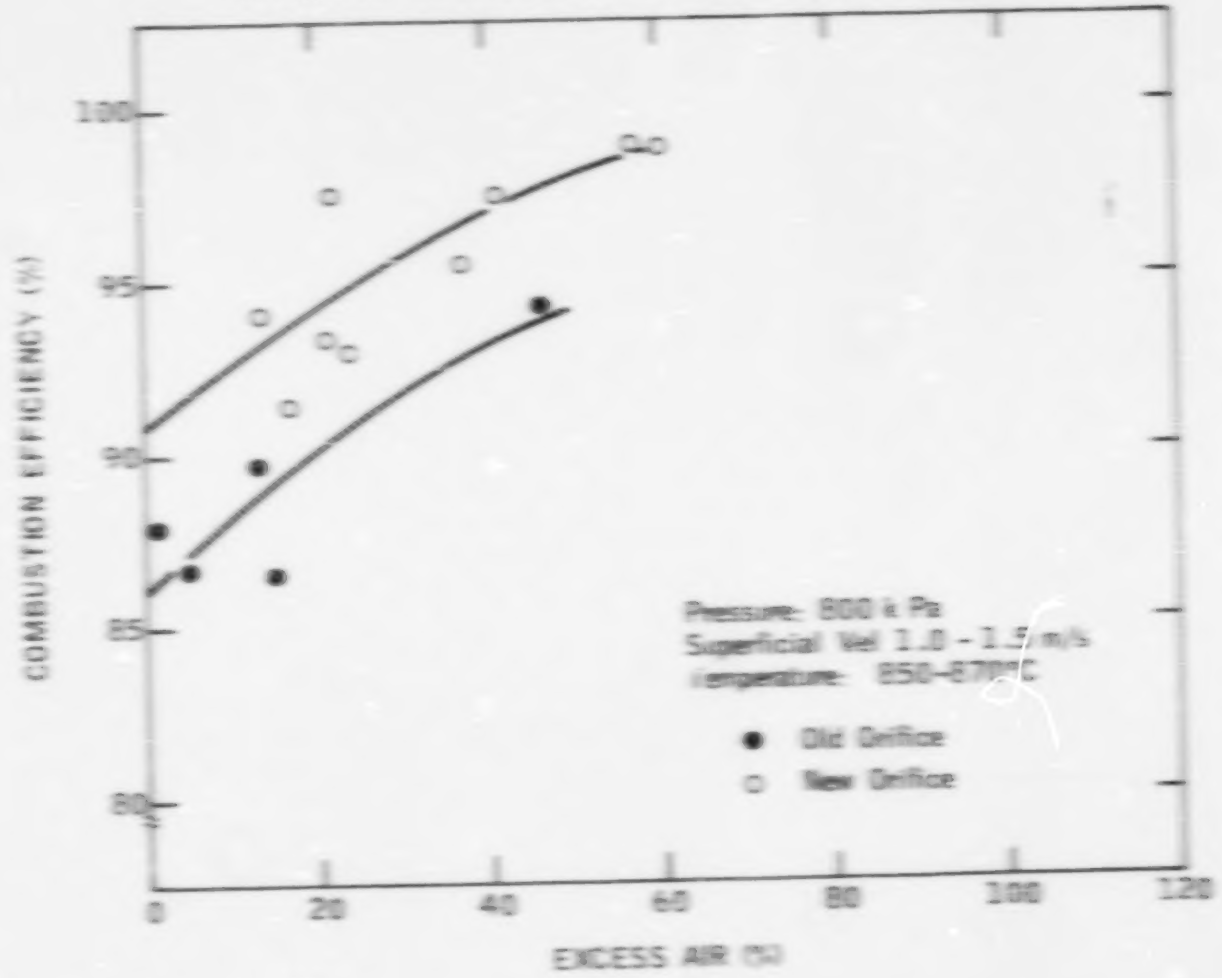


FIGURE III-43

EFFECT OF COAL FEEDING ON COMBUSTION EFFICIENCY



Particulate Emissions--

Particulate Loadings--Table III-26 presents a summary of the particulate loadings measured from batch unit runs 10-107C. Rejecting data which were greater than the mean by more than twice the error limit gave the following results:

TABLE III-26. PARTICULATE LOADINGS, BATCH UNIT

| Bed Material | Combustor Outlet | Grain Loading (gr/scf)<br>Primary Cyclone Outlet | Secondary Cyclone Outlet |
|---|---|---|---|
| Grove Limestone | 6.8 ± 1.4 | 0.9 ± 0.5 | 0.6 ± 0.5 |
| Tymochtee Dolomite | 7.0 ± 1.0 | 0.9 ± 0.5 | 0.4 ± 0.3 |
| Alundum | 5.3 ± 0.4 | 1.0 ± 0.5 | 0.3 ± 0.3 |

The data presented above show that the combustor outlet grain loadings were approximately the same for the limestone and dolomite runs. However, this is believed due primarily to differences in the operating conditions used during these runs such as superficial velocity, time of run, etc.

The overhead solids were routinely analysed for total carbon, total sulfur, sulfate, and carbonate. Appendix Table P presents a summary of these analyses. Using these results, it was calculated that ~20% of the overhead solids was elutriated bed material for the runs using Grove limestone and ~40% when Tymochtee dolomite was used, clearly indicating substantially higher attrition and entrainment rates for dolomite.

Particle Size Distribution--Overhead samples from selected runs were dry sieved to determine the particle size distribution. The results are presented in Appendix Table Q. The particle sizes for the 50% points were approximately 65 microns and 10-20 microns for the material collected by the primary cyclone and filter, respectively. These sizes are somewhat smaller than those previously reported.

Cyclone Efficiency--The average collection efficiency based on the average grain loadings calculated in the previous section was 86% for the primary cyclone and 46% for the secondary cyclone, giving an overall combined efficiency of 92%.

Components Balances--

Sulfur and calcium balances were made for a number of batch unit runs. The results are presented in Appendix Tables R and S. The $SO_2$ balances averaged 99.6% with a standard deviation of 20% and the calcium balances averaged 87% with a standard deviation of 10%. The weight of sulfur into the combustor was determined from the coal feed rate, the run length, and the sulfur content of the coal. The calcium input was determined from an initial bed analysis. For the solids output streams (material collected in the cyclone diplegs, off-gas filter, and the final bed), the quantity accumulated was weighed and then analyzed for sulfur, sulfate, and calcium. The quantity of sulfur in the flue gas was determined from data obtained by continuously monitoring the off-gas for $SO_2$.

# SECTION IV

# MINIPLANT REGENERATOR SHAKEDOWN

The shakedown of the miniplant regenerator system was carried out in two phases. The initial phase consisted in a series of shakedown runs made while operating the regenerator in a batch fashion, uncoupled from the combustor. The objectives of this phase were to test and develop equipment, gain operating experience and collect some data relating the $SO_2$ level in the regenerator off gas and sulfated sorbent conversion to the operating conditions. The second phase was aimed at demonstrating the continuous operation of the regenerator coupled to the combustor. The objectives of the second phase were to develop equipment required to move sorbent continuously between the combustor and regenerator, and again, to gain operating experience and collect data. This phase was to be concluded by the completion of a 24 hour continuous run.

This section describes the regenerator and the batch and continuous shakedown operations.

## EQUIPMENT

### Regenerator Vessel

The regenerator reactor, designed for operation at 1100°C and pressures up to 1010 kPa, consists of a 45.7 cm I.D. steel shell refractory-lined with 75-28 Grefco Litecast to an internal diameter of 21.6 cm. Numerous taps are provided along its 6.66 m overall height to monitor both temperature and pressure, while appropriately located ports allow for material entry and exit.

### Fuel System

There are two separate fuel systems: burner and supplementary fuel. Burner fuel is supplied to the burner where it is mixed with an approximately stoichiometric amount of burner air and burned. Supplementary fuel is added directly to the regenerator column just above the fluidising grid. Sufficient supplementary fuel is added to produce reducing gases (CO, $H_2$) at the desired concentrations. Location of the fuel (and air) inlets are shown in Figure IV-1.

Natural gas is compressed to about 180 psig by a Corken Model 590 compressor with a capacity of about 1.13 $m^3$/min (40 SCFM). Burner and supplementary fuel flow through separate lines, each containing a measuring orifice, automatic flow control valve, and an automatic shutoff valve. Supplementary fuel enters the regenerator just above the fluidising grid through a 0.64 cm (1/4 inch) O.D. stainless steel tube. A small flow of nitrogen is added to the supplementary fuel line when fuel flow is shut off in order to prevent bed solids from plugging the entry tube. Safety devices prevent addition of supplementary fuel into the regenerator until temperature is high enough for the fuel to burn satisfactorily, about 650°C.

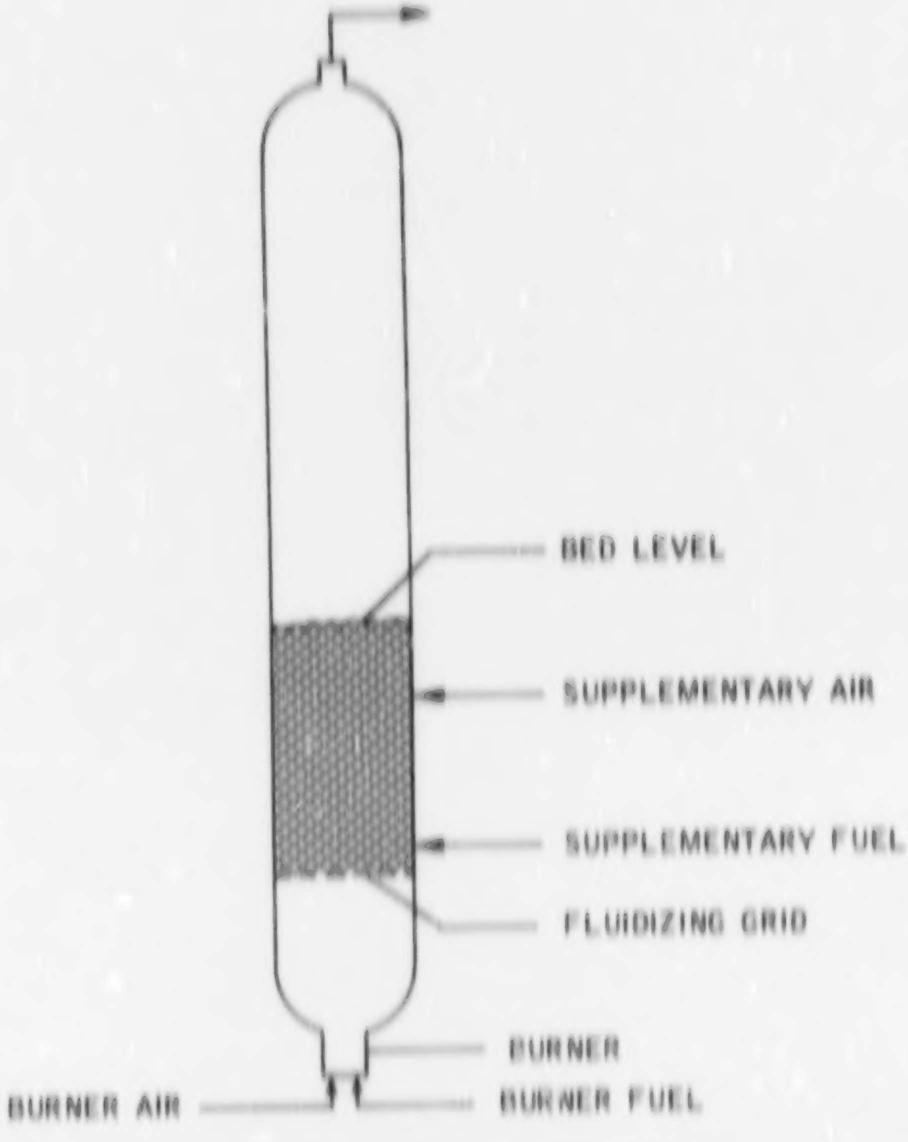

BED LEVEL
SUPPLEMENTARY AIR
SUPPLEMENTARY FUEL
FLUIDIZING GRID
BURNER
BURNER AIR
BURNER FUEL

FIGURE IV-1

MINIPLANT REGENERATOR AIR AND FUEL LOCATIONS

The ratio of flow rates of supplementary to burner fuel depends on the concentration of reducing gases desired; generally this ratio lies between 0.2 and 0.5.

### Air System

Two separate air systems can be identified on the regenerator: burner air and supplementary air. Burner air is supplied in sufficient quantity to burn completely the fuel (natural gas) supplied to the burner. Supplementary air is added directly to the bed in order to create an oxidizing zone in the upper portion of the bed.

The source of air for the regenerator is the main miniplant air compressor. Burner air can be made to flow through either of two measuring orifices, depending on the flow rates desired. Air is passed through a flow control valve tied to an automatic control loop and then enters the burner. Combustion products from the burner fluidize solids in the regenerator.

Supplementary air is also piped from the main air compressor through a measuring orifice and automatic flow control valve. It enters the regenerator through a 1.3 cm (1/2 inch) O.D. stainless steel tube whose outlet is positioned at the inside wall. Flow rates of supplementary air are typically about 20 percent of the burner air flow rate, but this can vary considerably depending on the air/fuel ratios desired in the oxidizing and reducing zones.

### Off-Gas Handling

Figure IV-2 shows the off-gas handling system for the regenerator. Gases leaving the regenerator are pressurized and hot (typically 900 kPa and about 930°C). The gas is cooled and pressure reduced before discharge into a wet scrubber. Principal components of the off-gas system are the cyclone, off-gas cooler, filter, pressure-control valve, and scrubber.

The regenerator cyclone removes the bulk of particulates, primarily entrained fines originating in the bed, from the gas stream. Gas inlet velocity to the cyclone is about 20 m/s, making it a moderately efficient cyclone. Off-gas then enters a cooler, which is a single pass double pipe heat exchanger 6.1 m long. The inner pipe, through which gas flows, is 3.8 cm (1-1/2 inch) Schedule 80 Type 316 stainless steel. Surface area for heat transfer on the inside of this pipe is 0.73 m$^2$. The outer pipe, through which water flows (either co-current or countercurrent) is 7.6 cm (3 inch) Schedule 40 steel pipe. Gases leave the cooler at 150-200°C, depending on the gas flow rate.

Before reducing pressure, dust is removed by passing the off-gas through a sintered stainless steel bayonet-type filter with a surface area of 0.14 m$^2$. Filtration is necessary to remove fine particulates which, if present in high concentrations, would erode the pressure reducing valve. This valve, a Norriseal Model 310 air-to-close with a $C_v$ = 6 orifice, is part of an automatic pressure control loop. Pressure is sensed at the top of the regenerator column with a Viatran pressure transmitter. Position of the pressure reducing valve is controlled by a Taylor electronic controller operating a Fairchild I/P converter.

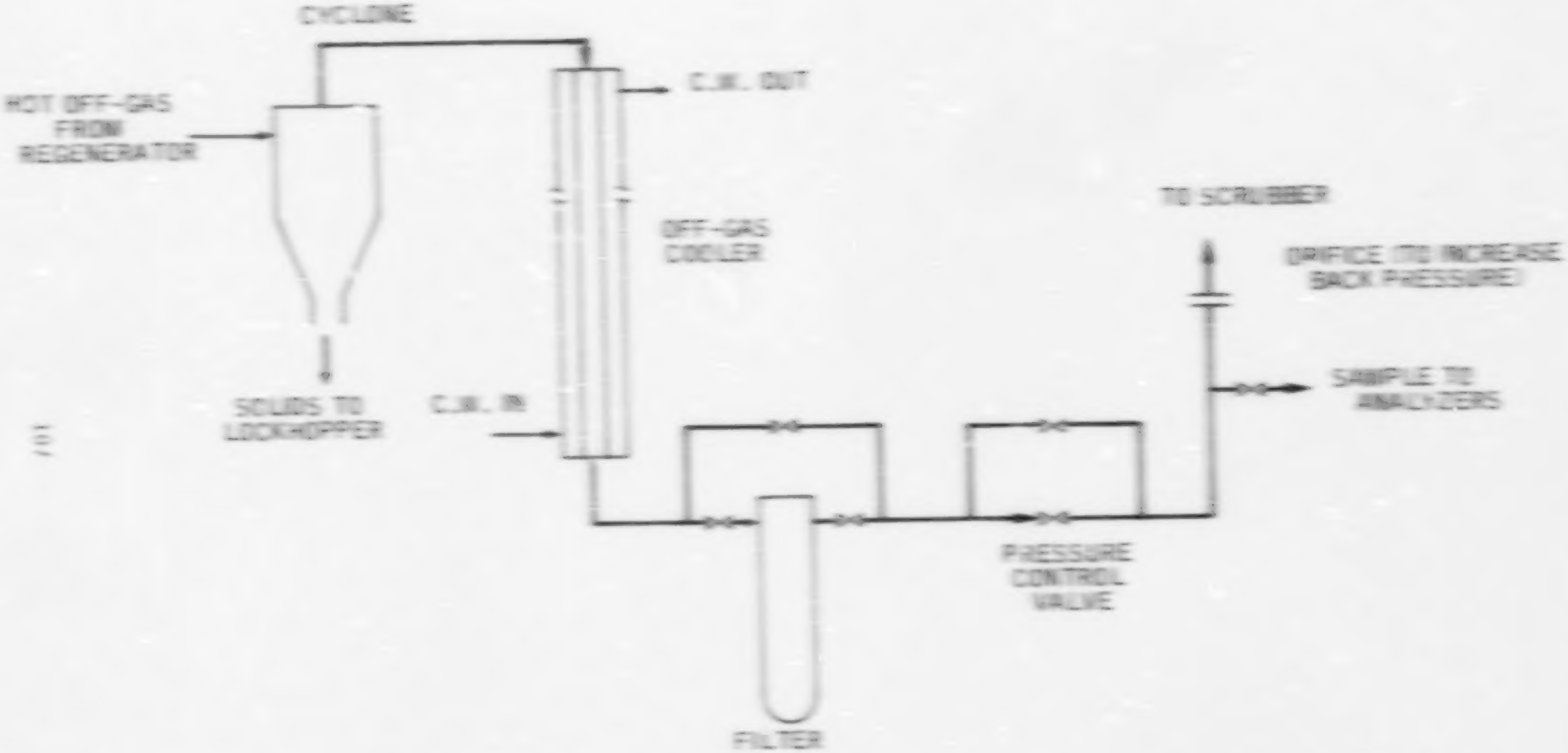

CYCLONE
HOT OFF-GAS FROM REGENERATOR
SOLIDS TO LOCKHOPPER
C.W. OUT
C.W. IN
OFF-GAS COOLER
FILTER
PRESSURE CONTROL VALVE
TO SCRUBBER
(ORIFICE (TO INCREASE BACK PRESSURE)
SAMPLE TO ANALYZERS

FIGURE IV-2

MINIPLANT REGENERATOR OFF-GAS HANDLING SYSTEM

Downstream of the pressure control valve, pressure of the off-gas is only slightly above atmospheric, and temperature is under 150°C. Regenerator off-gas is combined with off-gas from the combustor and piped to a Research Cottrell Scrubber for cleanup before venting. Sodium carbonate ($Na_2CO_3$) solution is added to the scrubber and reacts with the $SO_2$ to produce sodium bisulfite, $NaHSO_3$. An excess of $Na_2CO_3$ is used to keep the pH of the liquid in the scrubber in the range of 7-9. Scrubber liquid is slowly discharged into a diked area south of the miniplant. Calcium chloride ($CaCl_2$) solution is added to the liquid as it enters the diked area. Sulfite is precipitated as $CaSO_3 \cdot 2H_2O$ and excess $Na_2CO_3$ is precipitated as $CaCO_3$, thus keeping the pH of liquid in the diked area near neutral.

### Gas Sampling System

The system used to convey a sample of regenerator off-gas to analytical instruments is shown in Figure IV-3. The gas sample is obtained downstream of the pressure reducing valve so that the pressure of the sample gas is only slightly above atmospheric. A baffle located in the off-gas piping downstream of the sampling point serves to maintain the pressure of the sample stream high enough so that there is sufficient flow to the analyzers. Particulates are removed from the sample gas with a Balston Model 33 filter fitted with an "H" type filter tube. The clean gas then enters a Perma-Pure Model PD-1000-24S self-regenerative membrane type dryer. After leaving the dryer, the sample passes through Teflon lined tubing to the analyzer manifold. In order to prevent condensation of water, the sample line up to the dryer is maintained at a temperature of about 150°C.

A sampling point downstream of the pressure reducing valve was selected so that the sample could be obtained at low pressure. Sampling at low pressure reduces the residence time of gas in the lines, minimizing the possibility of gas composition changing before entering the analyzers. The need for a pressure regulator, which can corrode and change the sample composition, is also eliminated.

### Fluidizing Grid

The regenerator fluidizing grid, shown in Figure IV-4 is a stainless steel plate 13 mm thick into which are drilled 89 holes of 3.6 mm diameter. Because the grid is located directly above the burner, the grid must be water-cooled. Fourteen water channels, 4.8 mm in diameter, are located between the rows of holes to accomplish cooling. The size of 3.6 mm for the grid holes was chosen because this was about the largest size hole through which particles of bed material would not pass when the unit was shut down. The number of holes, which determines the total area available for flow of fluidizing gas, was chosen such that the pressure drop across the grid was about 25 percent of the bed pressure drop. This should result in uniform flow of fluidizing gas across the face of the grid.

### Burner

The burner used in the regenerator is identical to that used in the miniplant combustor. The burner was described in a previous report (2).

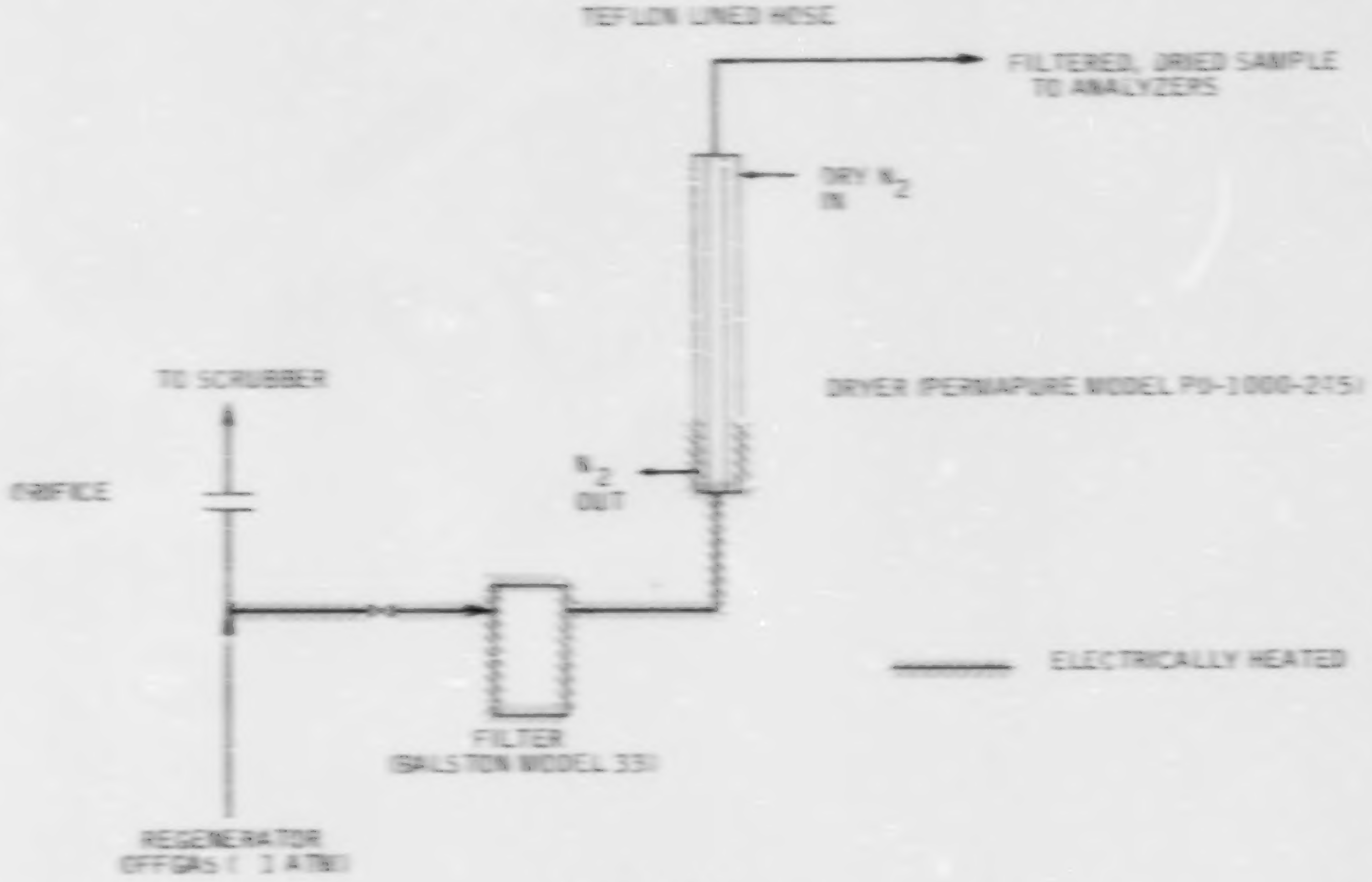

TEFLON LINED HOSE
FILTERED, DRIED SAMPLE TO ANALYZERS
DRY $N_2$ IN
DRYER (PERMAPURE MODEL PD-1000-24S)
TO SCRUBBER
ORIFICE
$N_2$ OUT
ELECTRICALLY HEATED
FILTER (BALSTON MODEL 33)
REGENERATOR (OFFGAS ( 1 ATM)

FIGURE IV-3

MINIPLANT REGENERATOR OFF-GAS SAMPLING SYSTEM

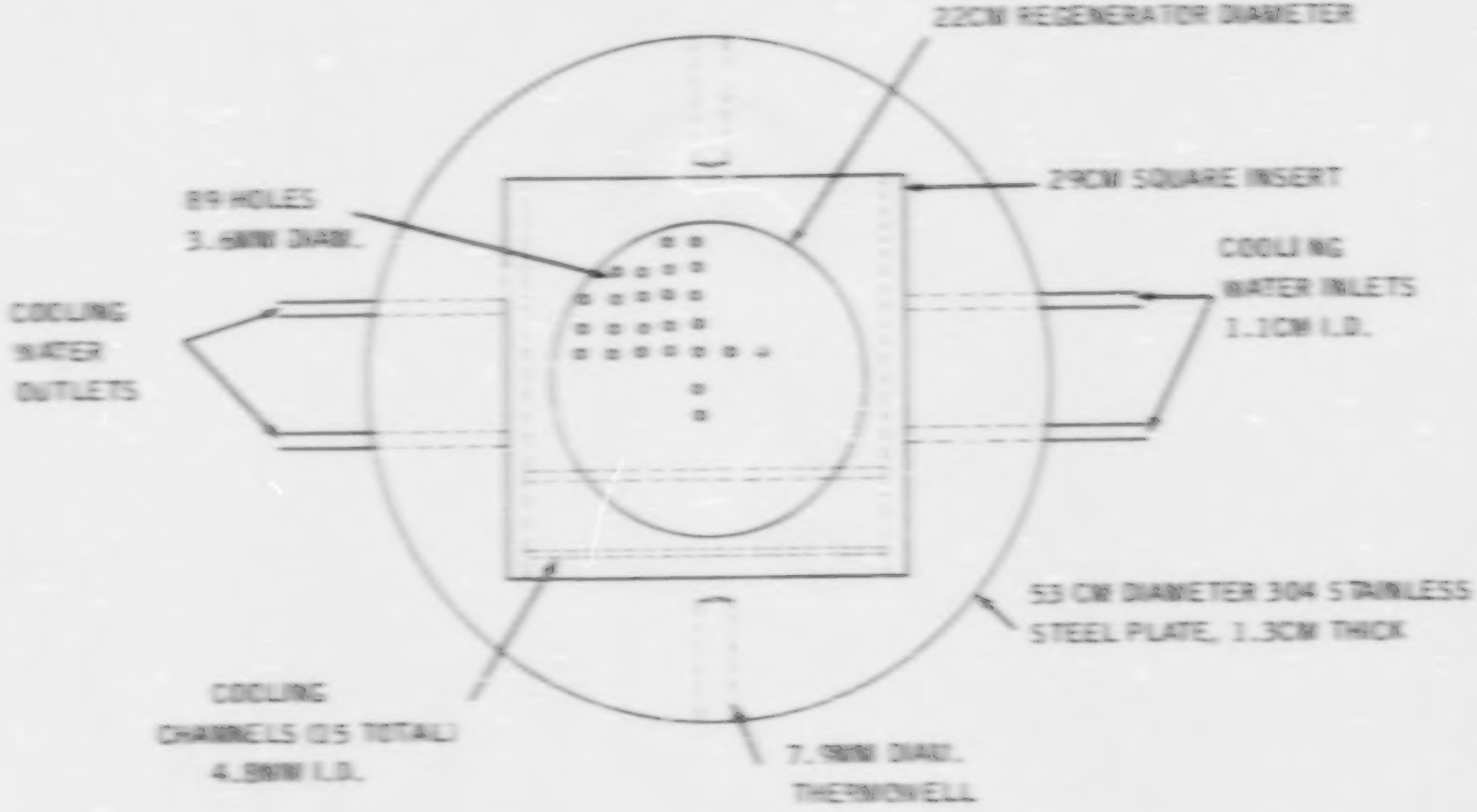

22CM REGENERATOR DIAMETER
29CM SQUARE INSERT
89 HOLES 3.6MM DIAM.
COOLING WATER INLETS 1.1CM I.D.
COOLING WATER OUTLETS
53 CM DIAMETER 304 STAINLESS STEEL PLATE, 1.3CM THICK
COOLING CHANNELS (25 TOTAL) 4.8MM I.D.
7.9MM DIAM. THERMOWELL

FIGURE IV-4

REGENERATOR FLUIDIZING GRID

# MATERIALS

## Materials of Construction

### Non-Metallic Materials--

*Refractory Lining*--The regenerator is constructed of 45.7 cm (18 inch) Schedule 40 steel pipe, refractory lined to an inside diameter of 21.6 cm. Thickness of the refractory insulation is about 10.6 cm. The refractory used is a castable type, General Refractories Litecast 75-28. This material has a service limit of 1540°C (2800°F) and a bulk density of about 1.2 g/cm$^3$ (75 lb/ft$^3$). Thermal conductivity at 540°C is 0.55 W/m$^2$°C (3.8 BTU/hr ft$^2$ °F/in).

Wear of the refractory lining, due to erosion caused by the bed and other factors, has not been estimated because of the relatively short time that the regenerator has been operated (less than 100 hours). No major obvious damage, such as cracking or spalling, has been noted as of July, 1979.

The section of the regenerator below the fluidizing grid (burner zone) is lined with General Refractories Bubbalite, which is also a castable, but with a temperature service limit of 1815°C.

*Thermocouple Protection Tubes*--The stainless steel sheathed type K (chromel-alumel) thermocouples used in the regenerator would fail quickly at the prevailing conditions of temperature and gas composition and therefore are protected in the following manner: The thermocouple is inserted into a 6.4 mm O.D. inconel tube which in turn is placed inside a silicon carbide sheath with an I.D. of 6.4 mm and an O.D. of 12.7 mm. The silicon carbide sheath provides excellent resistance to high temperatures and chemical attack and the inconel tube gives the assembly additional strength. Performance of this system has been fairly good although several silicon carbide tubes have cracked in service, probably from mechanical stresses.

### Metallic Materials--

*Off-Gas Cooler*--The off-gas cooler accepts hot (1000°C) pressurized gases from the regenerator and cools the gases to about 150-200°C. Process gases flow through a water-jacketed type 316 stainless steel pipe. The cooler is designed so that no condensation occurs during steady state operation; however, condensation cannot be prevented during startup and shutdown. Type 316 stainless steel offers good resistance to dry and wet $SO_2$ although wet $H_2SO_4$ can cause serious corrosion even at fairly low concentrations. Regular inspections of the cooler are made to check for the presence of corrosion. Thus far, with under 100 hours of actual operation, no corrosion has been detected.

*Filter*--A sintered stainless steel filter (bayonet type) is used to remove particulates from the off-gas before the gas passes through the pressure let-down valve. The filter element is made of Type 316 L stainless steel. The filter is sometimes wet, even under steady-state running conditions, and corrosion has been a problem. Corrosion has been minimized by thoroughly washing and drying the element immediately after the conclusion of each run. Allowing liquid to remain on the filter during downtime between runs resulted in very rapid corrosion. The off-gas cooler has recently been modified to provide higher temperatures in the filter, thus reducing condensation and corrosion problems.

The filter housing is made of Type 304 stainless steel. It is constructed of 10.2 cm (4 inch) pipe and is much more rugged than the commercially made housing that it replaced.

Gas Sampling System--The gas sampling system is shown in Figure IV-3. The sample is conveyed through a short length of 6.4 mm (0.25 inch) tubing into a Balston Model 33 filter. Both the tubing and filter are made of Type 304 stainless steel and both are heated to about 150°C to prevent condensation. Downstream of the filter the gas is dried and conveyed to analysers through Teflon lined tubing.

## Bed Material

All runs made in the regenerator have used sulfated stone prepared in the miniplant combustor. Three types of stone have been used: Grove limestone (No. 1359), Pfizer dolomite (No. 1337) and Tymochtee dolomite. Levels of sulfate in the bed charged to the regenerator varied from 15 to 30 weight percent.

## BATCH OPERATION

### Results

Eleven runs were made in which batch charges of sulfated limestone were regenerated. The purpose of these runs was to provide operating experience, test and develop equipment, and collect data. After the batch runs, a system to continuously transfer solids between combustor and regenerator was developed, installed, and tested, and continuous runs were carried out. Continuous runs are described in the section on Performance of Transfer System - 24 Hour Shakedown Run. This section describes the series of "batch" runs.

### Equipment Development

Supplementary Air and Fuel Systems--

From operation of the 8.3 cm diameter ("batch") regenerator in 1972-3, it was determined that improved performance could be obtained by adding supplementary fuel and air directly into the bed, in the manner shown in Figure IV-1 (1). The burner, located below the fluidising grid, is operated at an air/fuel ratio slightly above stoichiometric, and supplementary fuel is added just above the grid, in a quantity sufficient to produce a reducing atmosphere of the desired concentration (typically 65-85 percent of the stoichiometric air/fuel ratio). Higher in the bed supplementary air is added to bring the overall air/fuel ratio up to about stoichiometric.

Supplementary fuel (natural gas) compressed to about 1300 kPa flows through a measuring orifice, an automatic flow control valve, and 1.3 cm (0.5 inch) stainless steel tube to the regenerator. Fuel enters the bed through a 4.6 mm I.D. stainless steel probe that ends just at the inside wall of refractory. The probe was made a smaller diameter than the tubing which supplies fuel to the regenerator so as to impart a higher velocity to the fuel as it enters the bed. This provides better cooling for both fuel and probe, preventing overheating of the probe or decomposition of fuel in the probe.

Supplementary air is taken from the accumulator of the main air compressor. Flow control is obtained with an automatic valve which is part of a flow control loop. Flow rate is measured with an orifice and delta pressure transmitter. Air enters the regenerator through a 1.1 cm I.D stainless steel tube which terminates just at the inside refractory wall. Overheating of this tube has not been observed.

Only one position for the supplementary fuel inlet has been used; this is a point 12.7 cm above the fluidizing grid. Three positions for the supplementary air inlet have been tried: these have been at locations 58, 74, and 122 cm above the fluidizing grid. Height of the fluidized bed has typically been about 120-130 cm. Changing the locations of the supplementary air inlet changes the relative size of oxidizing and reducing zones. This might be expected to affect the amount of CaS produced in the bed and possibly the concentration of $SO_2$ that is generated; however, those effects were not observed. It should be noted that only one run was made with supplementary air admitted at each of the 58 and 122 cm locations, most work was done at 74 cm. No attempt was made to optimize the location as this would have required many more runs than time permitted. Moreover as the level of sulfide in the regenerated stone was already very low, not much benefit could result in this respect.

### Off-Gas Cooler--

The original off-gas cooler was a shell and tube unit approximately 1.8m long which contained nineteen 15.9 mm I.D. stainless steel tubes. Gas flow was through the tubes. Plugging of the tubes with solids was an occasional problem and the unit was replaced with the double pipe exchanger described on p.    . Performance has been satisfactory thus far, except for overcooling the off-gas under some operating conditions where the flow rate of off-gas was fairly low.

### Fluidizing Grid--

The fluidizing grid (air distributor) for the regenerator is a water-cooled stainless steel plate containing drilled holes for passage of the fluidizing gas. Cooling is essential because hot gases from the burner, located below, flow upwards through the grid.

The area available for flow of gases through the grid was chosen consistent with conventional practice, i.e., to produce a pressure drop equal to about 25% of the bed pressure drop. Reasonably uniform flow of gas over the face of the grid was therefore to be expected.

Two grids were used in the regenerator which differed primarily in the size and number of holes, but not very greatly in the total area for flow. The first grid, which was used for the first nine (out of eleven) runs made in the regenerator, contained 392 holes of 1.98 mm diameter. The second grid, shown in Figure IV-4, had 96 holes of 3.18 mm diameter. This was about the largest size hole through which solids would not flow when the unit was shut down. The new grid was cheaper to make because fewer holes had to be drilled; but, there was another possible advantage.

The type of grid can strongly influence the quality of fluidization and it is generally believed that a grid with many gas inlet openings is superior to one with few gas inlet openings because bubbles are smaller and gas-solid contacting is more intimate with many small openings. However, another school of thought contends that gas inlet openings should be larger than a minimum size. It is known that the penetration of a jet into a fluid medium increases as the diameter of the jet. Hence, larger gas inlet openings produce jets which penetrate further into the bed, stabilizing the bed and reducing the possibility of gross gas channeling. The new grid was designed with these considerations in mind.

Run no. 10 was the first using the new grid. During preheating, the temperature profile in the bed was flatter than had been seen before. After regeneration was started, the entire reducing zone of the bed, 74 cm high, was at the same temperature. The oxidizing zone of the bed, usually 10 to 50°C hotter than the reducing zone, was at the same temperature as the reducing zone.

During run no. 11, also made with the new grid but at far more severe reducing conditions, the temperature spread across the reducing zone was only 7°C. The temperature of the oxidizing zone was 11°C higher than the reducing zone. The unusual uniformity of temperature implies that the quality of fluidization was improved.

The $SO_2$ concentrations for runs 10 and 11, with the new grid, were 35% and 70% higher respectively, than the highest concentrations obtained previously (in run no. 8). It should be noted that these concentrations were 69% and 52%, respectively, of the concentrations that would be expected if chemical equilibrium had been obtained.

Changes in operating conditions are not suspected of causing the increase in $SO_2$ concentration. It appears that the change in grid design was responsible.

Thermocouple Protection Tubes--

The earliest thermocouple protection tubes used were silicon carbide sheaths which were slipped over the thermocouples. The thermocouples were Type K and were enclosed in a 3.2 mm O.D. stainless steel sheath. Castable refractory was then used to fill the entire carbide sheath in order to seal the thermocouple into the sheath. Breakage of these tubes occurred frequently, usually they were snapped in two.

Another approach was to flame spray protective coatings on the thermocouples. Chromium carbide was placed over a nickel aluminate substrate. However, these thermocouples failed quickly.

The third approach was to use again a silicon carbide sheath (6.4 mm I.D.) and to insert into the sheath an Inconel tube (6.4 mm O.D.) to provide reinforcement. The thermocouple was then placed in the Inconel tube. Although the silicon carbide sheaths have cracked occasionally, this method was by far the most satisfactory to date.

## Procedures

Startup--

Checkout of the regenerator and its support equipment is carried out prior to startup. This includes cleaning off-gas and sample filters, checking cooling water flows and setting manual valves in the air, fuel, and off-gas systems to proper positions. The nitrogen compressor is turned on so that flow is directed to all ports where pressure is measured, preventing these ports from plugging when bed is added to the regenerator column. About 50 kg of sulfated stone is usually added to the column, forming a settled bed of 1.0-1.3 m. Air and natural gas compressors are started and air is very slowly admitted to the column until pressure has reached about 200 kPa. The burner is usually ignited at pressures between 200 and 400 kPa. Heating of the bed is done slowly without increasing pressure, so that superficial gas velocity remains as high as possible (but always under 2 m/s). Bed temperatures during heatup are more uniform at higher velocities. When temperature approaches the desired level, generally 1040-1120°C, pressure is increased to 910 kPa (the pressure at which all regeneration runs were made). Sometimes small mounts of supplementary fuel are added to increase further bed temperature, but not enough to produce reducing conditions. Supplementary fuel can only be added when bed temperature is above 650°C, the temperature at which natural gas will ignite.

Normal Operating Procedures--

When the bed temperature is uniform and close to the temperature desired under reducing conditions, the switchover to reducing conditions is made. This is accomplished by increasing flow of supplementary air to the required value and then increasing the flow of supplementary fuel. Supplementary air flow is always increased before supplementary fuel so that air is not added to a column already filled with a reducing gas. Temperature is continuously monitored and flow rates of burner air, burner fuel, supplementary air, and supplementary fuel are all adjusted to yield the desired bed temperature (see section on Temperature Control). Oxygen and CO concentrations in the off-gas are also monitored and the supplementary air flow is corrected to produce low concentrations of CO (under 5000 ppm). The flow rate of supplementary air is the minimum value which will just produce CO in the oxidizing zone.

Shutdown--

Normal shutdown can be accomplished several ways. One method is to press the "emergency stop" button, which shuts all flows of air and fuel, fills the column with nitrogen and slowly depressurizes the column. This method is preferable if the final composition of solids is important, as nitrogen plus the rapid temperature drop "freezes" the composition of the bed.

Another method of shutdown is to turn off supplementary fuel and air flows, in that order. Burner fuel is shut next. Column pressure and burner air flow must be brought down together so as not to create high superficial velocities, which would blow solids out of the column.

Emergency Procedures--

During operation, shutdown is automatic if bed temperature exceeds 1260°C, if column pressure exceeds 1140 kPa, if burner air flow is lost, or if a burner flameout occurs. The operator can shut down the unit at any time by depressing a red emergency stop button on the control panel. An extensive system of alarms alerts the operator to out-of-limits conditions on about 20 variables.

Temperature Control--

The source of heat for regeneration is combustion of a fuel, natural gas. Heat sinks are (1) heating air and fuel entering the regenerator to temperature, typically about 1100°C; (2) endothermic heat of reaction for converting $CaSO_4$ to CaO, equivalent to about 1300 kJ/kg $CaSO_4$ converted; (3) sensible heat required to bring solids entering from combustor to regenerator temperature (applies to a continuous system) and (4) heat losses. Temperature of the fluidized bed is controlled by regulating the energy or fuel input. As shown in Figure IV-1, air and fuel are each added to both the burner and bed (supplementary air and fuel) in order to create adjacent oxidizing and reducing zones. Since it is usually desired to maintain fixed air/fuel ratios in these zones, it is necessary to adjust air flows whenever changing fuel flows. Thus, in order to change temperature, it is necessary to change the flow rates of all four streams: burner and supplementary air and burner and supplementary fuel. In practice, this is easy to do since all four flow rates can be changed automatically from the control panel.

It is important to maintain a well-controlled and uniform temperature in the regenerator because of the danger of agglomerating solids should localized temperatures get too hot (e.g., greater than 1150°C). Also, because the equilibrium concentration of $SO_2$ increases sharply with temperature, it is necessary to operate at as high a temperature as possible in order to obtain the highest $SO_2$ level.

Uniformity of bed temperature in the miniplant regenerator has generally been quite good. Variation of temperature within the reducing zone, which extends from the fluidizing grid to 74 cm above it, has been less than 10°C. The oxidizing zone is typically 10-50°C hotter than the reducing zone. The temperature difference between zones depends on the air/fuel ratios: with low air/fuel ratios in the reducing zone, more combustion occurs in the oxidizing zone, which results in higher temperatures. A temperature profile for a run made with the second fluidizing grid (larger holes) is given in Figure IV-5.

A problem exists in controlling temperature when the regenerator is run in "batch" fashion, i.e., when a batch of sulfated stone is charged and then regenerated. The problem arises because the heat required by the highly endothermic regeneration reaction varies throughout the run as the rate of reaction. Soon after reducing conditions are established the energy requirement rapidly increases and then gradually falls off as the regeneration rate decreases. The magnitude of the problem is illustrated in the following example. The energy balance can be represented as:

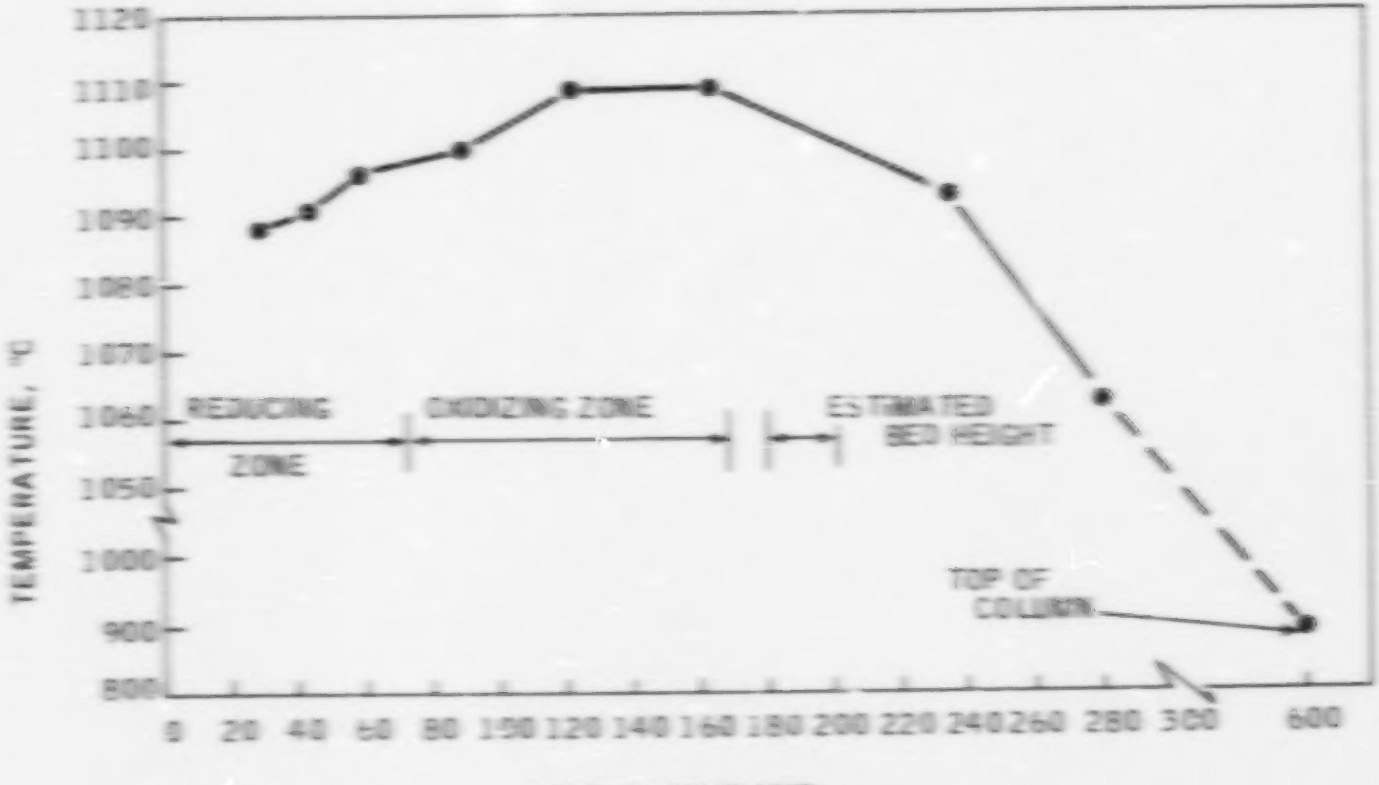
$$E = MC_p(T - Ta) + Q_{reaction} + Q_{losses} \qquad (1)$$

where $E$ = total energy input to regenerator
$M$ = mass flow rate of combustion gases
$C_p$ = heat capacity of combustion gases
$T$ = regenerator temperature
$Ta$ = temperature of entering air
$Q_{reaction}$ = endothermic heat of reaction
$Q_{losses}$ = heat losses

The first term on the right hand side of equation (1) represents energy required to heat gases to regeneration temperature. In a typical run, $Q_{reaction}/MC_p(T-Ta) = 0.40$, i.e., the average energy requirement for the regeneration reaction is about 40 percent of the energy required to heat

FIGURE IV-5

TYPICAL TEMPERATURE PROFILE IN MINIPLANT REGENERATOR BED



gases entering the regenerator to temperature. If regeneration should cease, and the energy input remains constant, the temperature will increase to T' according to:

$$1.40\ MC_p(T - Ta) + Q_{losses} = MC_p(T' - Ta) + Q_{losses}$$

$$\text{or } T' = 1.40T - 0.40Ta$$

For example, if T = 1100°C and Ta = 65°C, then T' = 1514°C. Therefore, bed temperature can increase over 400°C if energy input remained constant when regeneration ceased, as at the end of the run.

Practical consequences of the above problem are that the fuel input to the regenerator must be frequently adjusted as the regeneration rate peaks and then tapers off. In order to maintain constant bed temperature, total input of fuel to the regenerator had to be decreased by about 40 percent over the course of a typical batch run.

In continuous, steady, operation, where solids are transferred to and from the regenerator at constant rates, the above problem does not exist because the heat load on the regenerator does not vary. Regeneration proceeds at a fixed rate unless some external factor causes a change.

Bed Agglomeration--

Agglomeration of bed material was invariably associated with excessively high temperatures; when temperature was well controlled agglomeration did not occur. During the first few batch runs control problems caused air and fuel flow rates to be erratic, at times, and control of temperature was made especially difficult. Agglomeration did occur in moderate amounts, usually towards the end of the runs, when bed temperature would tend to increase because of a fall off in regeneration rates.

During a run at 1120°C, which was the highest temperature attempted, problems developed in controlling pressure and flow rates of air and fuel became unsteady, causing temperature at the supplementary fuel inlet to rapidly increase to about 1350°C even though the flow of fuel was shut down soon after the rise in temperature began. Examination of the bed revealed some agglomeration but the limited amount suggested that high temperatures were localized. Runs have been made at average bed temperatures(reducing zone) up to 1092°C without any agglomeration.

It has been observed that when localized high bed temperatures cause agglomeration to begin it is difficult to reverse the process and bring temperature back under control. Whenever agglomeration occurs, temperatures at different points in the bed quickly diverge and the portion of the bed that is already too hot becomes even hotter. This is not surprising because of the drop in the rate of heat transfer away from the agglomerated portion of the bed. Hence, agglomeration tends to be "autocatalytic;" once initiated, conditions become more favorable for further agglomeration.

Bed agglomeration appears to occur at temperatures considerably below the melting points of either pure $CaSO_4$ or CaO. It is known that the presence of even small amounts of coal ash reduce the temperature at which agglomeration has been observed to occur. This had been noted in early work carried out with pure $CaSO_4$ in the 8.3 cm diameter regenerator. Agglomeration occurred in runs made with sulfated limestone prepared in a coal combustor, but not in runs made with $CaSO_4$ which had not been exposed to coal (1).

$SO_2$ Levels--

The maximum $SO_2$ concentration that can be produced at a given temperature and pressure is the equilibrium concentration of $SO_2$ for the reaction:

$$3CaSO_4 + CaS = 4CaO + 4SO_2$$

This concentration is inversely proportional to total pressures and increases strongly with temperature. Table IV-1 gives partial pressures of $SO_2$ at several temperatures and the corresponding mole fractions at 910 kPa (nine atmospheres), the pressure at which runs were conducted.

TABLE IV-1. MAXIMUM PARTIAL PRESSURES OF $SO_2$ FOR REDUCTION OF $CaSO_4$

| Temperature °C | $SO_2$ Partial Pressure, kPa | $SO_2$ Mole Fraction at 910 kPa Total Pressure |
|---|---|---|
| 1000 | 21 | 0.023 |
| 1025 | 24 | 0.026 |
| 1050 | 27 | 0.030 |
| 1075 | 35 | 0.038 |
| 1100 | 52 | 0.057 |
| 1125 | 73 | 0.080 |
| 1150 | 106 | 0.116 |

$SO_2$ levels from regenerator runs made with batches of sulfated stone have been reported as average and peak values. Average values are somewhat ambiguous because they are influenced by the time chosen to end the run. Typically, $SO_2$ concentration increases rapidly at the outset, peaks, and then gradually tails off. The longer a run continues, the lower will be the average concentration of $SO_2$. Usually, runs were terminated when $SO_2$ levels declined to about ten percent of their peak values.

Because the off-gas is dried before analysis, measured concentrations of $SO_2$ and other components must be corrected. This correction is made as follows:

$$[SO_2]_{wet} = (1 - [H_2O])\,[SO_2]_{dry}$$

where $[H_2O]$ is the mole fraction of water present in the off-gas. Since this quantity is not measured it must be calculated by material balance.

Table IV-2 is a summary of runs made in the miniplant regenerator. $SO_2$ concentrations are reported as average and peak mole fractions and as the ratio of the peak $SO_2$ concentration to the equilibrium concentration of $SO_2$ expected at the temperature and pressure of the run. The temperature used in determining the equilibrium concentration is the temperature in the oxidizing zone of the bed, just before the gas reaches the bed surface. In some runs it was difficult to estimate average temperatures accurately; hence in these cases the ratio of measured to equilibrium $SO_2$ concentration is a rough approximation.

For ten runs, the mean of the average $SO_2$ concentration was 1.0 mole percent. The mean of the peak concentration was 1.8 mole percent. The mean of the ratio of peak to equilibrium $SO_2$ concentrations was 0.46, after eliminating three runs (nos. 2, 4, and 9) with very low ratios.

### Extent of Regeneration of $CaSO_4$ to CaO--

Two figures of merit have been developed to express the degree to which sulfated stone has been regenerated. The first, denoted as fractional regeneration, gives the fraction (0 → 1) of sulfur removed from the stone. In order for fractional regeneration to equal unity, all sulfate present in the original stone must be converted to oxide. The second figure of merit is called the fractional sulfide formation, and is the number of moles of sulfide produced per mole of sulfate decomposed. Sulfide formation must equal zero if fractional regeneration is equal to one.

The fractional regeneration and sulfide formation are computed from analyses of the initial and final stone (i.e., before and after regeneration) according to:

$$\text{regeneration} = \frac{\left(\frac{\text{w/o S}}{\text{w/o Ca}}\right)_i - \left(\frac{\text{w/o S}}{\text{w/o Ca}}\right)_f}{\left(\frac{\text{w/o S}}{\text{w/o Ca}}\right)_i}$$

$$\text{sulfide formation} = \frac{3\left(\frac{\text{w/o S}^{-2}}{\text{w/o Ca}}\right)_f}{\left(\frac{\text{w/o SO}_4^{-2}}{\text{w/o Ca}}\right)_i - \left(\frac{\text{w/o SO}_4^{-2}}{\text{w/o Ca}}\right)_f}$$

where w/o denotes weight percent, S refers to total sulfur and $S^{-2}$ to sulfide. All quantities in the above expressions are taken per unit weight of calcium in order to prevent the figures of merit from changing due to a reaction not involving sulfate or sulfide. For example, if only calcination occurred, then w/o S would increase because the stone loses weight; however, w/o S ÷ w/o Ca would remain the same.

To calculate the figures of merit expressed above, one requires concentration of Ca and $SO_4^{-2}$ in the initial stone, and Ca, $SO_4^{-2}$, $S^{-2}$, and total S in the regenerated stone. Initial stone contains sulfur only as sulfate so total S can be obtained from $SO_4^{-2}$. Unfortunately, all of this information

TABLE IV-1. REGENERATOR RUN SUMMARY

| Run No. | Charge | Settled Bed Height, m | Pressure kPa | Avg. Bed Temp., Red./Ox. Zones, °C | Sup. Gas Vel. Red./Ox. Zones, m/s | Air/Fuel Ratio in Reducing Zone (2) | $SO_2$ Conc. in Effluent Avg./Peak,(3) Mole % | Peak $SO_2$ Conc. / Equil. $SO_2$ Conc. |
|---|---|---|---|---|---|---|---|---|
| 1 | SG (1)<br>32 kg | 0.6 | 910 | 1120/1116 | 0.92/1.16 | 6.5 | not measured | |
| 2 | SG, 29.6 wt. % $SO_4$, 32 kg | 0.6 | 910 | 1065/1065 | 0.79/1.07 | 6.6 | 0.7/1.2 | 0.09 |
| 3 | Same as run 2, 48 kg | 0.9 | 910 | 1050/1054 | 0.72/0.91 | 6.0 | 0.6/1.2 | 0.39 |
| 4 | Same as run 2, 48 kg | 0.9 | 910 | 1070/1124 | 0.68/0.93 | 6.0 | 0.6/1.3 | 0.16 |
| 5 | ST, 19% $SO_4$, 48 kg | 0.9 | 910 | 1027/1088 | 0.75/0.85 | 6.6 | 0.5/1.4 | 0.30 |
| 6 | SP, 17.2% $SO_4$ 48 kg | 0.9 | 910 | 1077/1094 | 0.64/0.82 | 6.8 | 1.1/1.9 | 0.37 |
| 7 | SP, 17.2% $SO_4$ 48 kg | 0.9 | 910 | 1077/1088 | 0.73/0.77 | 8.3 | 0.8/1.7 | 0.36 |
| 8 | SG, 15.0% $SO_4$, 48 kg | 0.9 | 910 | 1071/1071 | 1.0/1.0 | 8.1 | 1.4/2.1 | 0.58 |
| 9 | SG, 25.8% $SO_4$, 48 kg | 0.9 | 930 | 1087/1112 | 0.93/0.96 | 7.8 | 0.4/1.3 | 0.19 |
| 10 | SP, 21.5% $SO_4$, 48 kg | 0.9 | 910 | 1063/1083 | 0.90/0.90 | 7.8 | 1.8/2.9 | 0.65 |
| 11 | SG, 37.5% $SO_4$, 48 kg | 0.9 | 910 | 1052/1105 | 0.84/1.07 | 6.7 | 2.0/3.0 | 0.52 |

Notes: (1) Codes for Charges:
SG = sulfated Grove limestone
ST = sulfated Tymochtee dolomite
SP = sulfated Pfizer dolomite

(2) Stoichiometric air/fuel ratio = 9.9

(3) wet basis, corrected for water condensed prior to analysis

is available only for the last six of eleven runs made in the regenerator. Furthermore, for many runs the total sulfur analysis was not consistent with the sulfate and sulfide analyses (1/3 w/o $SO_4^{-2}$ + w/o $S^{-2}$ should equal w/o S). In such cases, sulfide was calculated as the difference of total sulfur and 1/3 of sulfate since it was believed that analyses for these components (barium precipitation for sulfate and Dietert technique for total sulfur) were more accurate than that for sulfide (iodometric titration).

Table IV-3 gives fractional regeneration and sulfide formation for six runs. These results indicate that: (1) most sulfur was removed from the stone during regeneration and (2) formation of sulfide was near zero for all runs. The lower regeneration for runs 10 and 11 may be a result of stopping these runs earlier than the others.

TABLE IV-3. FRACTIONAL REGENERATION AND SULFIDE FORMATION

| Run No. | Regeneration | Sulfide Formation |
|---|---|---|
| 6 | 0.99 | 0.01 |
| 7 | 0.99 | 0 |
| 8 | 0.99 | 0 |
| 9 | 0.97 | 0.01 |
| 10 | 0.84 | 0.01 |
| 11 | 0.88 | 0 |

Note: Definitions of "Regeneration" and "Sulfide Formation" are given in text.

Material Balances--

Material balances for sulfur were calculated for the last six runs made in the regenerator. Results are given in Table IV-4.

Sulfur enters the system only in the sulfated stone that is charged prior to a run. Sulfur leaves in the off-gas as $SO_2$, in bed that is discharged from the regenerator after a run, and in fines that are entrained from the bed. A portion of the entrained fines are collected by a cyclone; however, fines that are not collected represent a loss of sulfur that is not accounted for in the material balance.

With the exception of two runs in Table IV-4, sulfur recoveries are quite low. There are a number of factors that could account for this; the most likely are (1) loss of bed when it is being removed from the regenerator after a run, (2) loss of sulfur in particles entrained from the bed and not collected by the cyclone, (3) errors in analyzing solids. The most serious potential cause of low sulfur recoveries would be erroneous determinations of $SO_2$ concentration in the off-gas, caused either by the gas sampling system or a malfunction of the analyzer. These latter possibilities appear unlikely because both the sampling equipment and analyzer had been thoroughly checked.

## TABLE IV-4. SULFUR BALANCES FOR REGENERATION RUNS

| Run No. | Sulfur In, kg | Sulfur Out, kg | | | % S Recovered |
|---|---|---|---|---|---|
| | Bed Charged | Bed Recovered | Dipleg Solids | Flue Gas | |
| 11 | 5.96 | 0.93 | 0.36 | 2.01 | 55 |
| 10 | 3.41 | 0.15 | 0.36 | 2.67 | 93 |
| 9 | 4.10 | 0.06 | 1.00 | 0.55 | 39 |
| 8 | 2.38 | 0.01 | 0.61 | 2.08 | 113 |
| 7 | 2.73 | 0.02 | 0 | 1.66 | 62 |
| 6 | 2.73 | 0.03 | 0.01 | 1.87 | 70 |
| 5 | 2.87 | 0.04 | 0.57 | 0.93 | 55 |

On the other hand, loss of solids collected after a run are known to be at least 10-20 percent, and possibly quite more for some runs. Calcium balances will be made in future runs in order to provide evidence that loss of solids is a major cause of the low sulfur recoveries.

$CO_2$ concentrations were calculated from air and fuel flow rates for each run and compared to measured values. For the last six runs, measured $CO_2$ concentrations averaged 86 percent of the calculated values, with a range of 76-105 percent. A reason why measured $CO_2$ concentrations are low may be that nitrogen is added at several places to the regenerator (e.g., for purges at pressure taps). This additional flow is not figured when $CO_2$ concentrations are calculated. An error in the air flow rate could also account for the difference between measured and calculated concentrations. These discrepancies will be further examined because they may also affect $SO_2$ concentration.

## Discussion

The batch runs made in the regenerator were extremely useful from the standpoint of establishing operating procedures and providing operating experience necessary to improve the performance and reliability of equipment. It was clearly shown that a batch of sulfated limestone or dolomite could be regenerated under well controlled conditions to produce a gaseous effluent containing about three mole percent $SO_2$. Furthermore, calcium sulfate could be converted almost completely to calcium oxide with very little formation of sulfide, using the technique of adjacent reducing and oxidizing zones.

A cause for optimism is that agglomeration of the bed could be avoided by careful control of temperature. In experiments made at Exxon several years ago in an 8.3 cm diameter regenerator (1) (the miniplant regenerator is 21.6 cm in diameter) most runs were made with pure calcium sulfate. A run in which real sulfated limestone was used resulted in a badly agglomerated bed. Also even though pure calcium sulfate was used in the smaller regenerator, only about two percent $SO_2$ was produced at temperatures under 1100°C. Three percent $SO_2$ was obtained in the last two runs made in the miniplant regenerator, using sulfated limestone. The improved performance of the miniplant regenerator, compared to the smaller unit, is probably largely due to the improved quality of fluidization in the miniplant. The best means available to determine how well solids are fluidized is the closeness of the temperature distribution in the bed. Temperatures in the miniplant were much more uniform, indicating that quality of fluidization was much better than in the smaller unit.

Peak values of measured $SO_2$ concentration were as high as 69 percent of the equilibrium concentration at the temperature and pressure of the run. The average approach to equilibrium was 46 percent. Temperature of the run refers to the temperature in the oxidizing zone, where gas contacts the last solids before leaving the bed.

Equilibrium concentrations were calculated based on the available free energy data for CaS, $CaSO_4$, CaO, and $SO_2$, assuming perfect gas behavior and no formation of solid solutions. However, Curran, et. al. (10), determined experimentally the equilibrium for the reaction:

$$3CaSO_4 + CaS = 4CaO + 4SO_2$$

and found that the equilibrium pressures of $SO_2$ were considerably lower than the calculated values. For example, at 1100°C, Curran's value is 37 kPa compared to 52 kPa for the calculated value.

Table IV-5 gives the ratio of the peak $SO_2$ concentrations for run Nos. 2-11 to the equilibrium concentrations determined by Curran. The ratios based on calculated $SO_2$ concentrations (given in Table IV-2) are repeated for comparison. In three runs (e.g., nos. 2, 4, 9) measured $SO_2$ concentrations were much less than equilibrium concentrations; however, abnormal conditions could account for this. In run nos. 2 and 4 the bed agglomerated and in run no. 9, a large portion of the bed was blown out of the regenerator.

Because of the considerable differences between the equilibrium $SO_2$ concentrations calculated from available free energy data and Curran's experimental values, additional experimental studies to find the true equilibrium would be worthwhile. If Curran's data are correct, then equilibrium was essentially established in several regeneration runs. If the calculated values are correct, then further improvement is possible toward increasing $SO_2$ concentrations.

## COUPLING OF REGENERATOR TO COMBUSTOR

The overall objective of this program was to demonstrate combined operation of the combustor and regenerator with circulation of solids between the two vessels. Characterization of the regenerative system will be made by operating the system over a range of test conditions. It is planned to determine what operating condition in the combustor and regenerator provide low emissions of $SO_2$ from the combustor, high concentrations of $SO_2$ in the off-gas from the regenerator, low makeup rates of fresh sorbent, and moderate recirculation rates between combustor and regenerator.

Problems were anticipated in developing a system to continuously transfer hot solids in the temperature range of 850-1125°C. Work proceeded in several stages: (1) cold-test alternative transfer systems to find that system which best deserved additional development, (2) make brief hot runs to further test the system and make improvements, (3) conclude shakedown of the combustor-regenerator by operating the units continuously for 24 hours at elevated temperature and pressure, (4) demonstrate long term operability by completing a run lasting 4-5 days, and (5) operate the combustor-regenerator over a range of test conditions to begin to characterize the system. This report describes stages (1)-(3), which were completed as of July, 1976.

### Equipment Development

#### Early Problems--

The first system tested depended on pulses of nitrogen gas to transfer solids. Figure IV-6 shows the combustor-to-regenerator transfer line. The regenerator-to-combustor line was similar. Solids from the combustor spill into the take-off port, fill the transfer line, and are moved into the regenerator by pulsing the bottom of the transfer line with nitrogen. The slide valve is used to correct upset conditions, for example to allow the line to be refilled with solids if they get blown out.

TABLE IV-5. COMPARISON OF MEASURED AND EQUILIBRIUM $SO_2$ CONCENTRATIONS

| Run No. | Temperature in Ox. Zone, °C | Peak Measured $SO_2$ Concentration, Mole % | Equil. $SO_2$ Conc. Calculated (1) Mole % | Equil. $SO_2$ Conc. based on Curran, (1) Mole % | $\frac{\text{Measured } SO_2}{\text{Calculated } SO_2}$ | $\frac{\text{Measured } SO_2}{\text{Curran } SO_2}$ |
|---|---|---|---|---|---|---|
| 2 | 1165 | 1.2 | 14 | 9.3 | 0.09 | 0.13 |
| 3 | 1054 | 1.2 | 3.1 | 1.7 | 0.39 | 0.71 |
| 4 | 1120 | 1.3 | 8.0 | 5.1 | 0.16 | 0.25 |
| 5 | 1088 | 1.4 | 4.7 | 2.9 | 0.30 | 0.48 |
| 6 | 1094 | 1.9 | 5.1 | 3.2 | 0.37 | 0.59 |
| 7 | 1088 | 1.7 | 4.7 | 2.9 | 0.36 | 0.59 |
| 8 | 1071 | 2.1 | 3.6 | 2.2 | 0.58 | 0.95 |
| 9 | 1112 | 1.3 | 6.7 | 4.2 | 0.19 | 0.31 |
| 10 | 1083 | 2.9 | 4.2 | 2.7 | 0.69 | 1.07 |
| 11 | 1103 | 3.0 | 5.8 | 3.7 | 0.52 | 0.81 |

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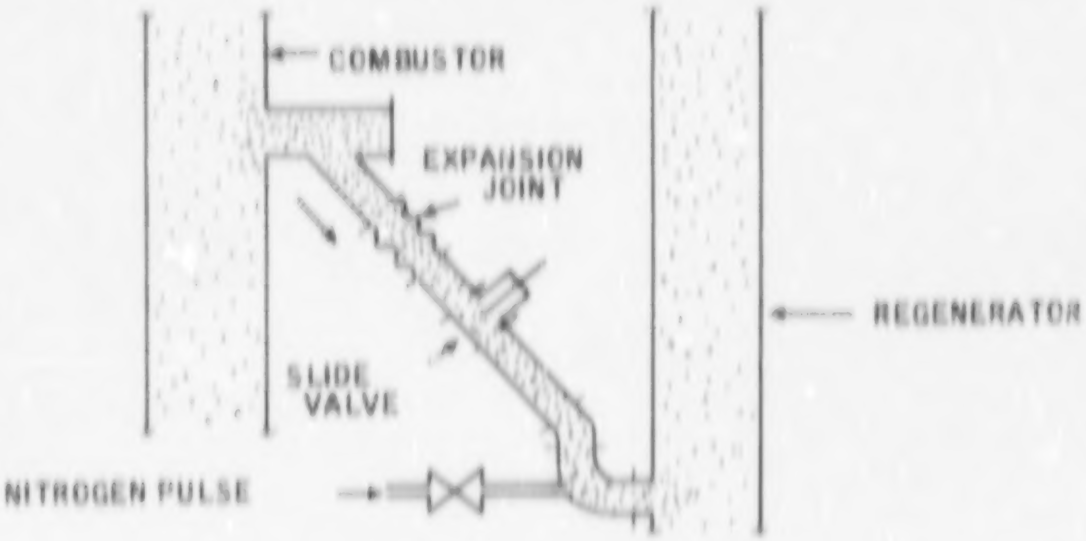
(1) For 9 atm total pressure and average temperature in oxidizing zone.



COMBUSTOR-REGENERATOR SOLIDS TRANSFER LINE

COMBUSTOR
EXPANSION JOINT
REGENERATOR
SLIDE VALVE
NITROGEN PULSE

FIGURE IV-6

ORIGINAL SOLIDS TRANSFER SYSTEM:
COMBUSTOR-REGENERATOR SOLIDS TRANSFER LINE

Early cold tests showed that solids could be easily transferred from combustor to regenerator provided that the level of the fluidized bed in the regenerator was low. As the bed level in the regenerator increased, pressure increased at the bottom of the bed, i.e., at the exit of the transfer line. This resulted in an increased back flow of gas up the transfer line. As the regenerator bed level became still higher, gas backflow increased sufficiently to blow solids up the transfer line.

Transfer in the opposite direction, from regenerator to combustor, followed a similar pattern. Only at low bed levels in the combustor could transfer be accomplished. This problem was explained by a simple pressure balance, shown in Figure IV-7. The pressure drop across the combustor-to-regenerator transfer line, $\Delta P_{12}$, becomes less favorable as bed level in the regenerator increases. The value of $\Delta Pa$ (regenerator pressure-combustor pressure) is the set point on the $\Delta P$ controller, and this value can be adjusted to provide a satisfactory value of $\Delta P_{12}$ for transfers from combustor to regenerator. However, $\Delta P_{34}$ would then be highly unfavorable for transfer from regenerator to combustor. Unfavorable values of $\Delta P_{12}$ or $\Delta P_{34}$ mean large negative values, large enough to permit excessive gas backflow.

It was concluded that by adjusting $\Delta Pa$ solids could be easily transferred but only one way at a time.

Modifications--

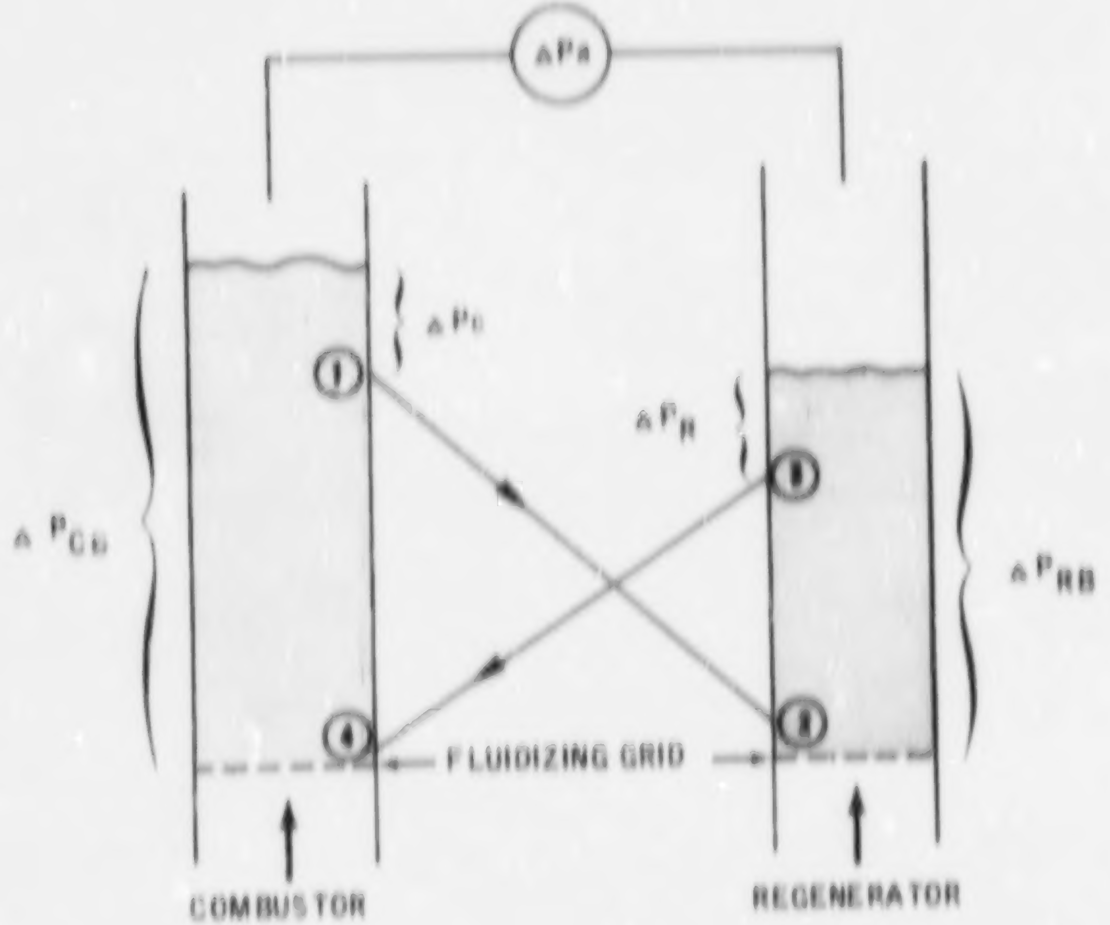
Several alternative approaches for transferring solids were proposed and cold tested. These would be used on only one transfer line; the second line would remain unchanged. The pressure balance would be adjusted so as to permit good transfer through the unmodified line. Summarized below are the alternatives that were investigated.

(1) Two Valves in Transfer Line - Solids would be trapped between the two valves by opening and then closing the upper valve. Transfer would be accomplished by pulsing or blowing out the solids after the lower valve was opened.

(2) Solids Reject Lockhopper/Pneumatic Conveying. Solids would be rejected and collected into a lockhopper in much the same manner as solids are rejected from the combustor. Solids would then be pneumatically conveyed from the lockhopper into the receiving column.

(3) Vary Combustor-Regenerator $\Delta P$. Solids can be transferred one way at a time by maintaining a favorable $\Delta P$ across the active transfer line. In this approach solids would be transferred from combustor to regenerator by setting the $\Delta P$ so as to facilitate transfer in this direction. This transfer line would then be closed with a slide valve and the $\Delta P$ adjusted to permit transfer through the regenerator to combustor line.

(4) U-tube - This technique is similar to that used in catalytic cracking to transfer catalyst between cracker and regenerator. Solids would spill into a long vertical standpipe where they would form an effective gas seal. Air (or nitrogen) would be added at the bottom of the standpipe to convey solids into the receiving column.


ΔPa
ΔPC
ΔPR
ΔPCB
ΔPRB
1
3
2
4
FLUIDIZING GRID
COMBUSTOR
REGENERATOR

$\Delta P_{12} = \Delta P_C - \Delta P_{RB} - \Delta P_a$

$\Delta P_{34} = \Delta P_R - \Delta P_{CB} + \Delta P_a$

WHERE $\Delta P_{12} = P_1 - P_2$, $\Delta P_{34} = P_3 - P_4$

$\Delta P_C$ = ΔP FOR COMBUSTOR BED ABOVE SOLIDS TAKEOFF

$\Delta P_R$ = ΔP FOR REGENERATOR BED ABOVE SOLIDS TAKEOFF

$\Delta P_{RB}$ = ΔP FOR ENTIRE REGENERATOR BED

$\Delta P_{CB}$ = ΔP FOR ENTIRE COMBUSTOR BED

$\Delta P_a$ = REGENERATOR PRESSURE − COMBUSTOR PRESSURE (MEASURED AT TOPS OF COLUMNS)

FIGURE IV-7

TRANSFER SYSTEM PRESSURE BALANCE

(5) Other Approaches. Two other possibilities include transferring solids up (rather than down) the transfer lines, or using a mechanical screw rather than pulsing gas to move solids into the receiving column.

Approaches (1)-(3) were cold tested and the transfer line lockhopper technique (2 valves in transfer line) was judged to offer the greatest potential for success. Figure IV-8 is a diagram of the piping configuration used in the transfer line lockhopper technique. Two automatic slide valves in the combustor-to-regenerator transfer line trap solids in the piping between them. Solids are discharged into the regenerator when the bottom valve is opened. A stream of purge nitrogen that is supplied to the valves assists in "pushing" solids out of the transfer line into the regenerator. Nitrogen pulses are supplied to the bottom of the combustor-to-regenerator line only as a backup method of keeping solids moving in the line. Pressure in the regenerator is maintained above the combustor so that the regenerator-to-combustor transfer line does not need two valves. The single manual slide valve in this line is closed only during startup and during upset conditions. Solids are blown into the combustor from the lower end of the regenerator-to-combustor line by applying a fairly strong pulse of nitrogen (producing a superficial velocity of approximately 2 m/s in the transfer line). The rate of solids transfer is controlled by the frequency of the pulse.

Two plugs in the solids takeoff ports are shown in Figure IV-8 in the open position. These plugs are inserted into the take-off ports during startup in order to prevent solids from entering the transfer lines. In the past, solids entering the transfer line during startup resulted in plugging of the transfer line when condensation of water caused the solids to agglomerate.

A six-hour test of the solids transfer system, during which coal was burned in the combustor, was completed successfully. Solids were transferred between combustor and regenerator at rates of about 70 kg/hr. No problems of any kind developed with the transfer system during the test. Combustor and regenerator operating conditions are summarized below. Illinois coal was fed to the combustor for about 1.3 hours; Champion coal was fed during the remainder. It should be noted that the regenerator was operated under oxidizing rather than reducing conditions. Reducing conditions could have been established by increasing the input of supplementary fuel. Operating the regenerator under reducing conditions would not have been expected to affect transfer of solids.

Operating Conditions During Six Hours Solids Transfer Test

| Variable | Combustor | Regenerator |
|---|---|---|
| Avg. bed Temperature, °C | 862 | 954 |
| Pressure, kPa | 870 | 870 |
| Superficial velocity, m/s | 1.8 | 0.73 |
| Expanded Bed Depth, m | 3.6 | 2.5 |
| Coal Feed Rate, kg/hr | 93 | -- |
| Excess Air, % | 49% | -- |

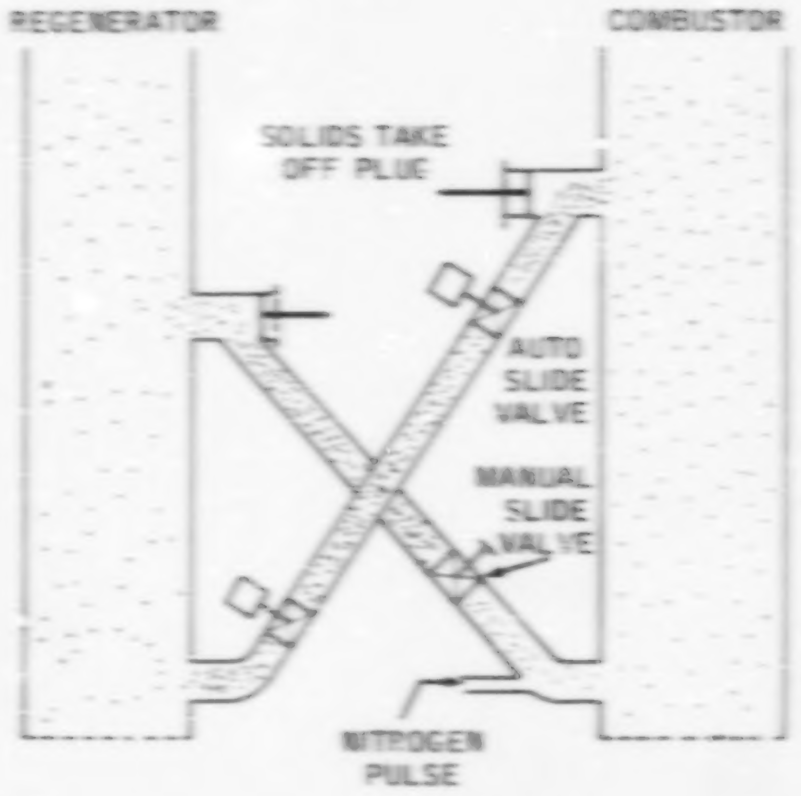

REGENERATOR
COMBUSTOR
SOLIDS TAKE OFF PLUG
AUTO SLIDE VALVE
MANUAL SLIDE VALVE
NITROGEN PULSE

FIGURE IV-8

MINIPLANT SOLIDS TRANSFER SYSTEM

Components of Transfer System--

Slide Valves--The slide valves are a critical part of the solids transfer system. They must be able to withstand hot solids at temperatures up to about 1000°C, working pressures up to 1000 kPa, and potentially corrosive gas atmospheres. Furthermore, the valves must be repeatedly open and shut. Because of these severe operating conditions, Exxon Engineering Technology Department (EETD) was asked to evaluate our design and recommend improvements. As a result, a modified design was developed which included (a) enlarging the bore of the valve from 5.1 to 7.6 cm, eliminating the stainless steel liner and replacing it with reinforced refractory, using a wider knife with a blunt edge and providing better cooling for the knife, and using a more rugged stem packing system.

Expansion Joints--Each transfer line contains an expansion joint to take up thermal expansion. These joints were quite bulky and made it difficult to perform mechanical work on the transfer lines. EETD was therefore asked to determine if expansion joints were really needed. Their analysis showed that the joint on the combustor-to-regenerator line could be removed if skin (shell) temperatures were under 177°C; the joint on the regenerator-to-combustor line could be removed if temperatures were under 135°C. The cumbersome expansion compensators attached to the joints were judged not needed and were removed.

Measurements of transfer line skin temperatures were made during hot tests. Not surprisingly, skin temperatures depended on solids' transfer rates. For rates up to about 145 kg/hr maximum average skin temperatures for the combustor-to-regenerator and regenerator-to-combustor transfer lines were 146°C and 200°C, respectively. During these tests, the regenerator was at 980°C, a somewhat lower than normal temperature. The combustor temperature was about 900°C. Hence, it appears that the expansion joint on the regenerator-to-combustor line will be required, but that the joint on the combustor-to-regenerator line could be removed.

Transfer Line Pipe and "Pulse Pots"--The transfer lines are fabricated from 15.2 cm (6 inch) Schedule 40 pipe refractory lined to an inside diameter of 7.6 cm. The sloping portions of the transfer lines are sleeved with 6.4 cm (2-1/2 inch) Sch. 10 316 stainless steel pipe with an inside diameter of 6.7 cm. Because the slide valves have a smaller diameter bore (5.1 cm) than the lines, transition pieces are used to reduce smoothly the diameter from 6.7 to 5.1 cm.

Pulses of nitrogen which are admitted to the lower section of the regenerator-to-combustor transfer line, called a "pulse-pot," blow solids lying between the pulse-pot and regenerator vessel into the vessel. Nitrogen enters through a 1.3 cm (1/2 inch) stainless steel tube whose outlet is positioned approximately at the center of the pulse-pot. Superficial velocity of nitrogen in the 7.6 cm diameter line connecting the pulse pot and regenerator is about 2 m/s. The solids transfer rate is controlled by adjusting the duration and frequency of the pulse. Typically, a pulse lasting 1-2 seconds occurs every 30-60 seconds.

## Procedures

### Startup of Transfer System--

The solids transfer system is not operated during startup to prevent moisture from entering the transfer lines. Plugs are inserted in the solids takeoff ports so that solids cannot spill into the lines from the combustor and regenerator vessels. Nitrogen is pulsed into the transfer line pulse pots in order to prevent solids from backing up into the lines. The slide valves are also cycled occasionally in order to dislodge any solids that may have passed by the takeoff plugs. Pressure in the regenerator is set slightly above that in the combustor (usually 2.5-10 kPa) and the beds are heated to near operating temperatures. Transfer of solids is started by pulling the plugs out of the take-off ports.

### Operation--

Transfer rate is controlled by setting the cycle time of the slide valves in the combustor-to-regenerator line. The valves are opened and closed automatically by a series of timers on the miniplant control panel. Action follows the following sequence: top valve opens, line fills with solids, top valve closes, bottom valve opens, line empties, bottom valve closes. The volume between the slide valves holds about 6.0 kg of solids; hence, setting the cycle time gives the transfer rate of solids from combustor to regenerator. If bed levels are kept constant, then the transfer rate from regenerator to combustor must be the same.

### Shutdown--

It is important, during shutdown, to empty the transfer lines of solids; otherwise plugging of the lines may occur when the unit is restarted. Hence, the first step is to shut the plugs in the solids takeoff ports, thereby preventing solids from entering the lines. Cycling of the slide valves in the combustor-to-regenerator line and pulsing nitrogen in the regenerator-to-combustor line is continued until the miniplant is shutdown so that the lines are emptied.

## Performance of Transfer System -
## 24 Hour Shakedown Run

The miniplant was operated continuously for 24 hours in order to test the system for transferring solids between the combustor and regenerator. Operation of the system is described and process data are given in the following sections.

### Operation of the Solids Transfer System During Shakedown Run--

The miniplant regenerator and combustor were operated simultaneously for 24 hours on July 26-27, 1976. Solids were continuously transferred between the two vessels for the entire period. Operation of the solids transfer system went extremely well and not a single problem developed once flow of solids was begun. The regenerator was operated under reducing conditions for about 23 and 1/2 hours. However, problems developed in feeding Illinois coal and kerosene had to be burned in the combustor for about six of the 24 hours. However, the basic intent of the run was to demonstrate continuous operation of the combustor and regenerator for a 24-hour period and this was done. Nominal operating conditions are given in Table IV-6. A log of noteworthy events during the run is given in Table IV-7.

TABLE IV-6. NOMINAL OPERATING CONDITIONS FOR COMBUSTOR AND REGENERATOR DURING SHAKEDOWN RUN

| | Combustor | Regenerator |
|---|---|---|
| Pressure, kPa | 910 | 915 |
| Temperature, °C | 900 | 1040 |
| Fluidized bed height, m | 4.5 | 2.5 |
| Superficial gas velocity, m/s | 1.8 | 0.7 |
| Combustor coal feed rate, kg/hr | 80.3 | -- |
| Combustor excess air, % | 44 | -- |
| Air/fuel ratio in regenerator reducing zone* | -- | 8.2 |
| Limestone feed rate to combustor, equiv. Ca/S | 0.8 | -- |
| Solids recirculation rate, kg/hr | 65 | |

---

* Stoichiometric = 9.9

TABLE IV-7. LOG OF EVENTS FOR 24 HOUR SHAKEDOWN RUN

| Hours After Run Start | Event |
|---|---|
| 0 | Coal feed started to combustor (Champion, 2% S) |
| 1/2 | Solids transfer begun from combustor to regenerator |
| 1 | Solids transfer begun from regenerator to combustor |
| 1 1/2 | Regenerator in reducing conditions |
| 14 1/2 | Plugging in coal feeding system due to Illinois coal, switched to kerosene fuel for combustor |
| 20 1/2 | Arkwright coal (2.6% S) feed started to combustor |
| 25 | Failure of regenerator fluidising grid, combustor and regenerator shut down |

Transfer rate of solids was 65 kg/hr during the entire run. The slide valves in the combustor-to-regenerator line were cycled every eight minutes. In the regenerator-to-combustor line nitrogen was pulsed for 1 second every 60 seconds.

Process Data - Shakedown Run--

It was intended to operate the combustor and regenerator at constant conditions, varying only the ratio of Ca/S in the feed in order to keep emissions of $SO_2$ from the combustor steady at about 300-400 ppm. However, steady conditions were not reached until about seven hours into the run. Over this period, sulfation levels in the combustor and regenerator reached approximately steady values. The material charged into the combustor and regenerator prior to the run was sulfated, not fresh, limestone. During a second seven-hour period after steady conditions were reached, $SO_2$ levels of 300-400 ppm were maintained with a Ca/S feed rate of 0.8. The level of $SO_2$ expected for once-through (non-regenerative) operation of the combustor at this Ca/S ratio was about 1000 ppm. This result showed that the regenerator was effectively reducing the emissions of $SO_2$ from the combustor or, put another way, decreasing the feed rate requirement of limestone into the combustor.

Emissions from both combustor and regenerator are summarized in Table IV-8. The regenerator may have also acted as a calciner. Conditions in the combustor favored carbonate whereas higher temperatures in the regenerator favored oxide. Hence, in a regenerative system it would be possible to use limestone in the combustor even at conditions where limestone would not calcine in the combustor.

TABLE IV-8. SHAKEDOWN RUN NOMINAL EMISSION LEVELS FROM COMBUSTOR AND REGENERATOR

| | Combustor | Regenerator |
|---|---|---|
| $SO_2$ | 350 | 8750 |
| $CO_2$, % | 14.0 | 13.5 |
| CO, ppm | 120 | 1825 |
| $O_2$, % | 6.3 | 0.3 |
| $NO_x$, ppm | 110 | 3 |

(For steady period, 7-14 hours from start of run.
Operating conditions given in Table IV-6)

The concentration of $SO_2$ in the regenerator off-gas was typically 0.7-1.0%. This level of $SO_2$ is close to that which is predicted by a sulfur material balance on the regenerative system. The equilibrium level of $SO_2$ at regenerator conditions (1040°C, 915 kPa) is about 2.8%. Hence, the $SO_2$ level was limited by the rate of input of sulfur to the system (material balance) rather than by thermodynamics. Increasing the coal feed rate or burning coal with a higher sulfur content could have increased the $SO_2$ concentration.

After 14-1/2 hours into the run (see Table IV-7), kerosene (which contains no sulfur) replaced the Champion coal feed. Regeneration continued during the six hour period during which kerosene was burned in the combustor. When coal combustion was resumed the measured level of $SO_2$ emissions in the combustor was zero, perhaps indicating that a fully regenerated and a highly calcined stone with a strong affinity to capture $SO_2$ was produced in the regenerator during this period.

Another optimistic result was that bed levels remained nearly constant for about the last ten hours of the run even though no makeup limestone was added. This may be due to a high degree of attrition resistance. More data will be needed, however, in order to determine the effect of cycling between combustion and regeneration conditions on the attrition resistance of the sorbent.

# SECTION V

# DISCUSSION OF RESULTS

## COMPARISON OF BATCH UNIT AND MINIPLANT RESULTS

The operation of the batch unit and miniplnat are quite different, and it is interesting to compare the more significant results from the two units. The miniplant is a continuous unit. A mixture of coal and sorbent is fed continuously and solids are removed directly from the bed to keep the bed height at a desired level. Some solids are also removed overhead as entrained flyash. The miniplant can be operated under steady state conditions. In the batch unit, on the other hand, only coal is fed continuously into a bed of sorbent whose composition changes continuously.

The sulfation level of the bed in the batch unit increases continuously, whereas, in the miniplant combustor, the average sulfation level of the bed reaches a constant value after a sufficient length of time on stream. There is always a certain amount of fresh sorbent in the miniplant bed giving a distribution of particles with different levels of sulfation. In the batch unit, all particles have the same "age" and approximately the same sulfation level.

Figure V-1 is a comparison of the sulfur dioxide retention in the miniplant and batch unit for runs with dolomite sorbent. The solid lines represent data from the miniplant for residence times of 1 second and 0.5 second. The points are retentions in the batch unit for residence times between 0.5 and 1 second. The retention in the batch unit is lower than would be predicted from miniplant data.

This result is not at all surprising when the difference between the two units is considered. It is known that a sorbent particle loses activity rapidly as its level of sulfation increases. Since there is a continuous stream of fresh sorbent into the miniplant, the activity of the bed is expected to be higher, for the same average level of sulfation, than that of the batch unit.

Figure V-2 shows a comparison of the combustion efficiencies of the two units. The solid lines represent two of the levels of combustion efficiency found in the miniplant. The points in Figure V-2 represent runs in the batch unit.

At lower temperatures the miniplant has a higher combustion efficiency than does the batch unit. However, data from both units seem to converge to greater than 99% at a temperature of about 975°C. The higher efficiency of the miniplant is to be expected since the unit is equipped to recycle overhead solids. This recycle of unburned carbon should increase the combustion efficiency.

Figure V-3 compares the $NO_x$ emissions of the two units. In general, the batch unit has higher emissions than does the miniplant. As pointed out earlier, the batch unit seems to give the same type curve of $NO_x$ vs. excess

FIGURE V-1

COMPARISON OF SULFUR DIOXIDE RETENTION MEASURED IN MINIPLANT
AND BATCH UNITS - DOLOMITE SORBENT

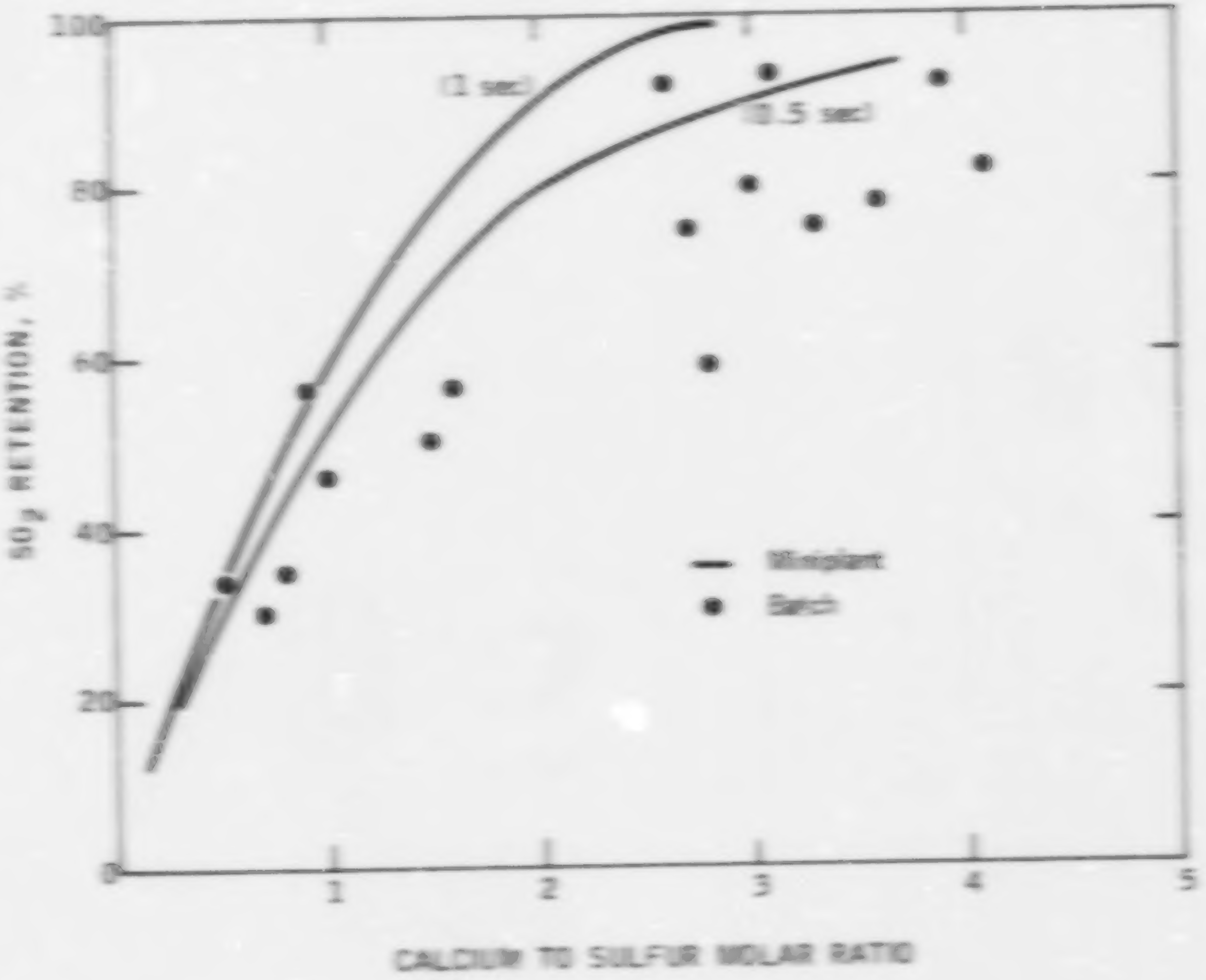


FIGURE V-2

COMPARISON OF COMBUSTION EFFICIENCIES MEASURED IN
MINIPLANT AND BATCH UNITS

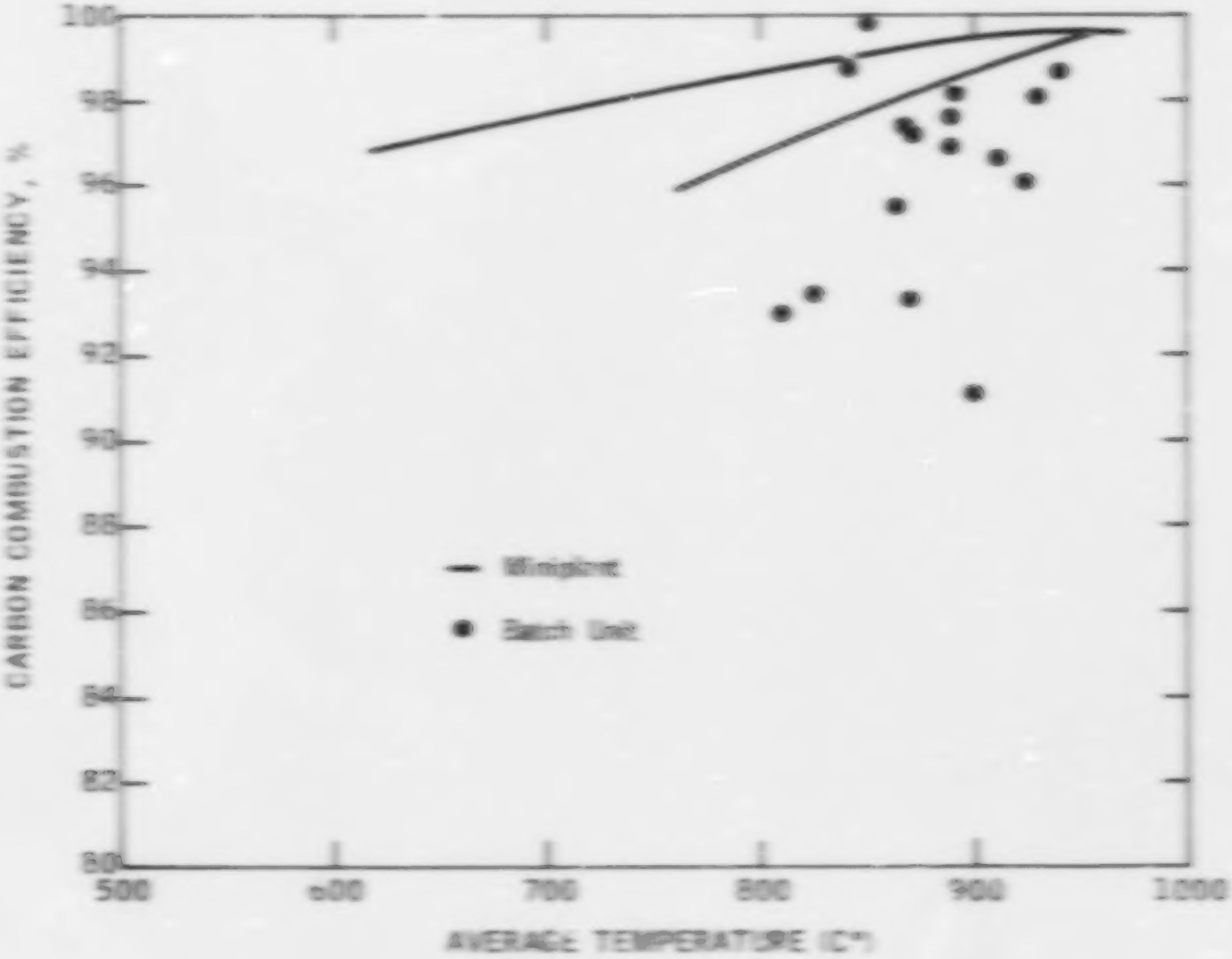
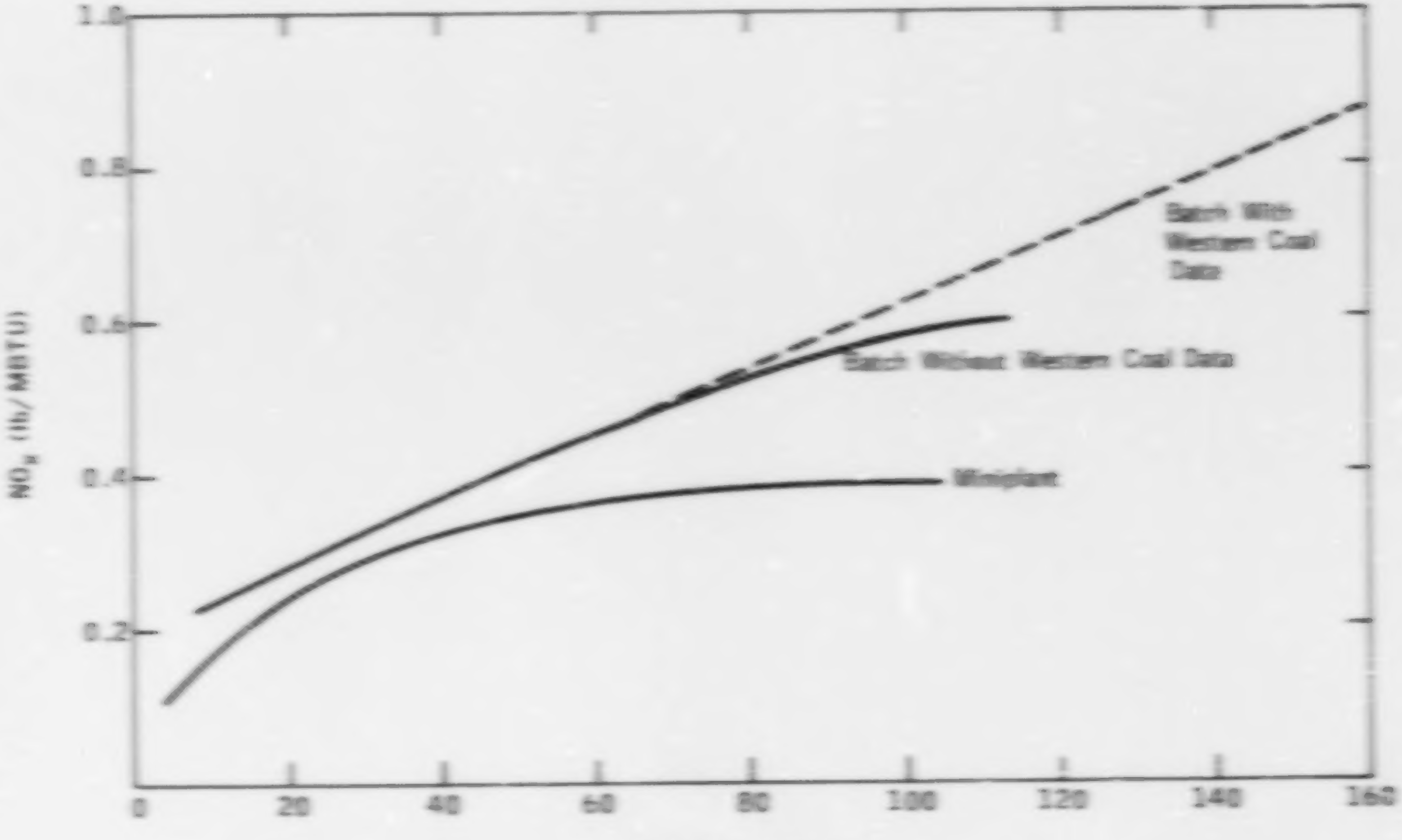


FIGURE V-3

COMPARISON OF $NO_x$ EMISSIONS MEASURED IN
MINIPLANT AND BATCH UNITS



air as does the miniplant when runs with Western coal are not included. If Western coal data are included, the $NO_x$ emissions from the batch unit continue to increase even to 160% excess air instead of leveling out as had been observed from miniplant results.

The difference in the $NO_x$ emissions from the two units is not easily explained. The nature of $NO_x$ formation and disappearance is not known. At the low temperatures of fluid bed combustion, it seems certain that the $NO_x$ is produced from the nitrogen in the coal. Various suggestions have been made as to how it disappears (e.g., reacts with CO, C, $SO_2$ , etc.) but since these mechanisms are not very well substantiated, explaining differences between two units is very difficult.

## SECTION VI

## CONTINUING STUDIES

The future program in the miniplant and batch units will be centered on four major tasks; combustion studies, flue gas particulate removal, a comprehensive analysis of emissions and a study of sorbent regeneration.

### COMBUSTION STUDIES

In the miniplant, the current combustion program will be continued to include tests using a high sulfur Illinois No. 6 coal with dolomite sorbent in a series of runs in which combustor temperature and Ca/S molar ratio are varied.

A study will also be made of the effect of using precalcined limestone sorbent under combustion conditions which do not favor extensive calcination of the limestone. If successful, this would increase the desulfurization activity of limestone at intermediate combustion temperature conditions where the stone is only partially calcined and the limestone activity is low. It could also make it possible to use limestone at very low temperature conditions, such as those occurring during "turndown" operation, where limestone does not calcine and is virtually inert.

A combustion program will also be carried out in the batch unit. The batch combustor will first be modified to convert it to a continuous unit. This will require the addition of continuous sorbent feeding and spent sorbent removal systems. The combustor will then be used in a program to provide technical support for the miniplant and also to investigate specific technical problem areas.

### FLUE GAS PARTICULATE REMOVAL

Particulates must be removed from the combustor flue gas to low levels to meet both environmental and gas turbine requirements. The current EPA particulate emission standard for a new coal fired boiler is 0.1 lb/M BTU of coal fired (0.043 g/MJ). Currently, particulate size and/or composition are not specified in the emission control regulations. For a typical coal, this standard translates to a particulate concentration in the flue gas of about 100 mg/m$^3$ (0.05 gr/SCF). This level cannot be reached by the use of conventional cyclones to reduce the particulate loading. A realistic level that can be attained by the use of two conventional cyclones operated in series is about 300 mg/m$^3$ (0.15 gr/SCF). Therefore if a two stage cyclone system is used to remove a portion of the particulates from the flue gas, a third stage device would still be required to reduce particulate loadings from the vicinity of 300 mg/m$^3$ to 100 mg/m$^3$ to meet current environmental standards. This requires a removal efficiency of about 67% in the third stage cleanup device.

Allowable particulate loadings to minimize gas turbine erosion are presently indicated to be even lower than those required to meet the environmental regulations. At the present time, turbine erosion limits are not well defined. Westinghouse Research Laboratory recently estimated allowable levels based on limited data and model studies (5). The estimates covered a range of 50 to 0.9 mg/m$^3$ (0.02 to 0.0004 gr/SCF). Based on these estimates, Westinghouse suggested a tentative allowable level of 5 mg/m$^3$ (0.002 gr/SCF). To meet this very low particulate concentration, a third stage particulate removal device would be required to operate at an efficiency of 98.7%. These allowable particulate concentrations and required removal efficiencies are summarized in Table VI-1.

TABLE VI-1. PARTICULATE EMISSION CONTROL REQUIREMENTS

| Requirement | Allowable Level (mg/m$^3$) | Allowable Level (gr/SCF) | Required Efficiency of Third Stage Device (%) |
|---|---|---|---|
| Environmental | 100 | 0.05 | 67 |
| Turbine Erosion | | | |
| Range of estimates | 50-0.9 | 0.02-0.0004 | 87-99.7 |
| Tentative level | 5 | 0.002 | 98.7 |

In addition to the above, an even tighter limitation on allowable particulate concentrations may be imposed by turbine corrosion considerations. However, at the present, insufficient data are available to estimate the allowable level required to prevent corrosion.

The objective of the flue gas particulate removal program is to evaluate two removal devices which have the potential for reducing the particulate loadings to the required levels. Since gas turbine considerations now appear to set the required particulate removal efficiencies, these more stringent efficiencies will form the target levels for the study. Although the primary intent of this EPA sponsored program is to measure and characterize the particulates escaping the third stage device for their potential environmental impact, the measurements, to be realistic, must be made under conditions which are aimed at protecting the gas turbines. The effect of the particulates on gas turbines is of secondary importance to this program and will be measured using an erosion test passage designed and fabricated by Westinghouse Research Laboratory under contract to the EPA. In addition, EPA and the Energy Research and Development Administration (ERDA) are cooperating on a second program to evaluate in the miniplant the resistance to erosion and corrosion of a number of turbine test specimens to be provided by the General Electric Company under contract to ERDA. Therefore, the particulate control program will ultimately be concerned with both environmental and gas turbine considerations.

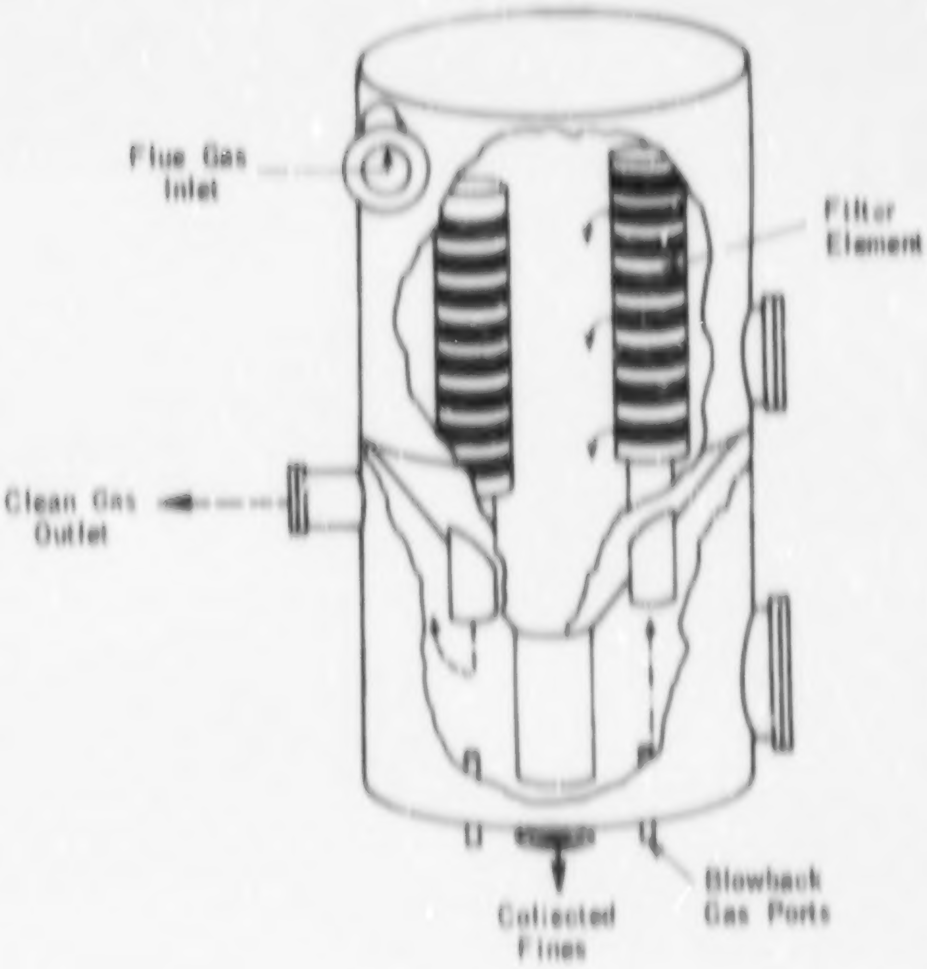
The first particulate control device to be evaluated will be a granular bed filter purchased from the Ducon Company. This filter was chosen after surveying the type of devices currently available. Use was made of a series of earlier surveys made by Stone and Webster (11) and Westinghouse (5). Discussions were also held with EPA and Exxon personnel active in the area of particulate control. Other control devices mentioned in the surveys are high temperature metal or ceramic filters and a low temperature scrubber combined with an efficient heat exchanger. However, the consensus favored the granular bed filters as the type of device which currently offers the best chance of meeting the high removal efficiency targets.

A number of groups are currently developing granular bed filter systems which are described in the above referenced Westinghouse and Stone and Webster reports. The Ducon Company has tested granular bed filters on refinery and other waste gas streams. A type of filter, called the panel bed filter is currently being studied at the City College of New York under sponsorship of the Electric Power Research Institute (EPRI). The Rexnord Company has installed a number of "gravel bed" filters on cement kiln and other industrial off gases. Combustion Power Company has also installed a number of "dry scrubber" filters on flue gas from wood waste boilers. Combustion Power is also developing a similar system for application on FBC.

The Ducon filter was chosen for testing on the Exxon/EPA miniplant after considering all the available systems. The selection was based on previous experience with the Ducon system which indicated that it had the potential of providing high removal efficiency. Another desirable feature of the Ducon system is the retention of the granular filter medium in the filter vessel. In all other systems, the medium is removed, cleaned externally and recycled back to the filter vessel.

A sketch of the conventional Ducon filter is shown in Figure VI-1. The filter consists of a series of beds containing the filter medium, stacked vertically to form a filter element. A number of elements are contained in a pressure shell. Dirty gas passes through an inlet screen, through the filter medium and out through an outlet screen into a central collecting tube. Clean gas exits the vessel at the bottom. Each element is periodically cleaned by a short pulse of high pressure clean air flowing in reverse flow through each element. The dust is blown out through the inlet screens and collects in the bottom cone of the pressure vessel. The filter medium is retained in the beds.

The filter system to be used on the miniplant will be a modified version of the conventional Ducon system described above. Four filter elements will be used and will be designed to test three different filter medium cleaning (blow back) techniques. Each element will be encased in a shroud to allow segregation of the particulates removed from it during the blow back. Separate feed ducts will supply dirty gas to each element and each duct will contain an orifice to permit measurement of flow to each element. One of the elements will use the high pressure pulse blow back described above. Another element will use a "positive blow back" technique. It will be equipped with shutoff valves to allow it to be completely isolated from the feed and product streams during blow back. The element can then be depressured and blown back with a larger volume of low pressure air. Two of the elements will use a modified "positive blow back" technique. In this case only one end of the element will be shut off by a valve during blow back to permit blow back by a larger volume of air slightly above the filtration pressure. The positive blow back systems should provide more effective regeneration of the filter beds and a better chance of meeting the high removal efficiency targets.


Flue Gas Inlet
Filter Element
Clean Gas Outlet
Blowback Gas Ports
Collected Fines

FILTER ELEMENT INTERNALS

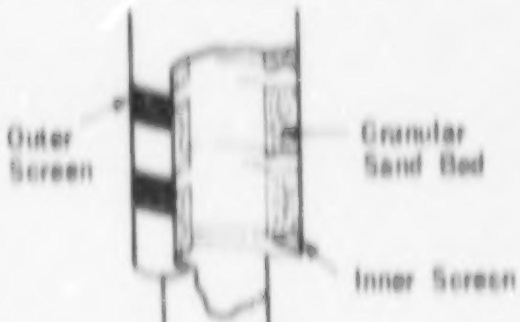

Outer Screen
Granular Sand Bed
Inner Screen

FIGURE VI -1
DUCON GRANULAR BED FILTER

The positive blow back element consist of 10 beds, each 0.3 m (1 ft) in diameter. The element is 1.8 m (6 ft) long. The remaining three elements contain 12 beds, each 0.3 m (1 ft) in diameter. These elements are also 1.8 m (6 ft) long. The elements are contained in a refractory lined pressure vessel designed for 1000 kPa (10 atm) operating pressure. The system will be capable of filtering 343 $m^3$/min (1200 SCFM) of hot flue gas. Installation is scheduled for completion in late 1976.

The filter will be evaluated and performance optimized in an experimental program in which the particulate removal efficiency and efficiency maintenance will be measured as a function of operating parameters. These parameters include the method of blow back, blow back frequency, duration, pressure and blow back gas volume. The nature and particle size of the filter medium will be varied as will the flow rate and particulate loading of the inlet gas. Provisions are included to permit major modifications to this filter system as required to improve and/or maintain particulate removal efficiency.

In addition to the evaluation of the Ducon filter, a second particulate removal device will be chosen, procurred, and tested. The choice of this device will be made in cooperation with the EPA.

## COMPREHENSIVE ANALYSIS OF EMISSIONS

A comprehensive analysis of gaseous and solid streams leaving the miniplant and batch units will be made to obtain a complete inventory of potentially harmful emissions. Sampling and analytical systems will be chosen for this program in cooperation with the EPA and Battelle Columbus Laboratory, the EPA Environmental Assessment Contractor for fluidized bed combustion.

The comprehensive analysis program will be carried out in three phases. In the first phase, "Level 1" analyses will be made to obtain an indication of the type of compounds present in the combustor flue gas before and after the third stage particulate removal device, in the regenerator off gas and in the solids streams removed from the combustor, i.e., the spent sorbent removed from the combustor and the particulates removed by the cyclones and third stage device. The particulates may be divided into two size fractions, respirable and non-respirable. The general type of chemical compounds to be sought in the sampled streams are various sulfur compounds, nitrogen compounds, organic compounds, inorganic carbon compounds, chlorine and fluorine compounds and trace elements. Particulates will also be tested for size, size distribution and physical structure. Biological testing of some of the sampled materials will also be carried out.

After Level 1 analyses have been conducted, Level 2 analyses will be made in which potentially harmful species identified in the Level 1 series will be measured quantitatively. This will be followed by Level 3 analyses which will determine periodically during operation of the fluidized bed units the concentration of those species identified in Level 2 as being present in significant concentrations.

## REGENERATION STUDIES

Regeneration studies will be carried out in miniplant and modified batch units. In the miniplnat, an extended demonstration run of 4 to 5 days duration will be made in which the combustor and regenerator will be operated continuously.

A series of regeneration tests will then be made in both the miniplant and batch units. In the batch unit, a test program will be run to identify the operating conditions which provide high levels of sulfur removal from the sorbent and high levels of $SO_2$ in the regenerator off gas. Cyclic studies involving the batch combustor will also be made to measure activity maintenance of the sorbent.

In the miniplant, a series of tests will be made which will be aimed at characterizing and optimizing the continuous combustion/regeneration system. The test series will determine which combustor and regenerator operating conditions provide, simultaneously, low emissions of $SO_2$ and other pollutants from the combustor, high concentrations of $SO_2$ in the regenerator off-gas, low makeup rates of fresh sorbent and moderate recirculation rates between combustor and regenerator. An additional objective will be to minimize the attrition rate of sorbents, since this will reduce the required amount of makeup sorbent and reduce the load on the flue gas particulate removal devices.

## SECTION VII

## REFERENCES

1. Hoke, R. C., et al, "A Regenerative Limestone Process for Fluidized-Bed Coal Combustion and Desulfurization," EPA-650/2-74-001, January 1974.

2. Hoke, R. C., et al, "Studies of the Pressurized Fluidized-Bed Coal Combustion Process," EPA-600/7-76-011, September 1976.

3. Skopp, A., et al, "Studies of the Fluidized Lime-Bed Coal Combustion Desulfurization System," Esso Research and Engineering Company for EPA, 1971.

4. Jonke, A. A., et al, "Reduction of Atmospheric Pollution by the Application of Fluidized Bed Combustion," EPA-Argonne National Laboratory, ANL/ES-CEN-1002, June 1970.

5. Keairns, D. L., et al, "Fluidized Bed Combustion Process Evaluation - Phase II Pressurized Fluidized Bed Coal Combustion Development," Westinghouse Research Laboratories, EPA-650/2-75-027c, September 1975.

6. Vogel, G. J., et al, "Annual Report on a Development Program on Pressurized Fluidized Bed Combustion," ANL/ES-CEN/1011, July 1975.

7. National Research Development Corp., "Pressurized Fluidized Bed Combustion," R&D Report No. 85, Interim No. 1 for Office of Coal Research.

8. Cox, D. G., et al, National Coal Board Final Report, "Reduction of Atmospheric Pollution," Vol. 2, for EPA, September 1971.

9. Vogel, G. J., et al, "A Development Program on Pressurized Fluidized Bed Combustion," Quarterly Report to U.S. Energy Research and Development Administration, Contract No. 14-32-0001-1780, January 1976.

10. Curran, G. P., Fink, C. E., and Gorin, E., "$CO_2$ Acceptor Gasification Process," Fuel Gasification, Advances in Chemistry Series, 69, 1967.

11. Zabolotny, E. R., et al, Stone and Webster Engineering Corp., Report to EPRI, November 1974.

# SECTION VIII

## LIST OF PUBLICATIONS

1. Hoke, R. C., Bertrand, R. R., "Pressurized Fluidized Bed Combustion of Coal," Institute of Fuel Symposium, Series No. 1: Fluidized Combustion Vol. 1. London, September 1975.

2. Hoke, R. C., "Emissions from Pressurized Fluidized Bed Coal Combustion," Proceedings of the Fourth International Conference on Fluidized Bed Combustion, Washington, DC, December 9-11, 1975.

3. Hoke R. C., "FBC Particulate Control Practice and Future Needs: Exxon Miniplant," Symposium on Particulate Control in Energy Processes, San Francisco, May 11-13, 1976.

4. Nutkis, M. S., "Operation and Performance of the Pressurized FBC Miniplant," Proceedings of the Fourth International Conference on Fluidized Bed Combustion, McLean, Virginia, December 9-11, 1975; pp. 221-238.

5. Ruth, L. A., "Combustion and Desulfurization of Coal in a Fluidized Bed of Limestone," _Fluidization Technology_, v. II, D. L. Keairns, ed., Hemisphere Publ. Corp., Washington, DC 1976, pp. 321-27.

6. Ruth, L. A., "Regeneration of CaSO$_4$ in Fluidized Bed Combustion," Proceedings of the Fourth International Conference on Fluidized Bed Combustion, McLean, Virigina, December 9-11, 1975; pp. 425-38.

7. Nutkis, M. S., "Pressurized Fluidized Bed Coal Combustion," _Fluidization Technology_, V. II, D. L. Keairns, ed., Hemisphere Publ. Corp., Washington, DC 1976, pp. 329-337.

## PATENT MEMORANDA SUBMITTED

1. Ruth, L. A., Design of Gas Distributors for Small Fluidized Beds.

# SECTION IX

## APPENDICES

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## TABLE 6. ANALYTICAL TECHNIQUES

### Analysis of Solids

Solids from combustion runs were analyzed for $SO_4^{-2}$, $CO_3^{-2}$, $Ca^{+2}$, $Mg^{+2}$ carbon and total sulfur. The analytical techniques that were used are described below.

$SO_4^{-2}$ = The sample was treated with acidic $BaCl_2$ solution. The $BaSO_4$ precipitate was weighed.

$CO_3^{-2}$ = HCl was added to an acidified sample. The solution was stripped with $N_2$ and the gas passed through drierite, $CuSO_4$ and ascarite. $CO_3^{-2}$ was determined from the weight gain of the ascarite.

$Ca^{+2}$ $Mg^{+2}$ = The sample was digested by heating vigorously in a medium of perchloric acid/nitric acid. The determination of Ca and Mg was made by atomic absorption.

Total Sulfur = The sample was mixed with sodium peroxide and a catalyst. The sulfur was converted to the sodium sulfate. The sample was heated above the melting point and the melt was extracted with water. The sulfur was converted to barium sulfate, precipitated and weighed.

Carbon = Samples were combusted within a packed tube in an oxygen atmosphere. Helium was used to sweep the combustion gases into the analytical system. Carbon dioxide was determined by differences in thermal conductivity.

### Analysis of Flue Gas by Wet Chemical Methods

$SO_3$ = The amount absorbed by an 80% isopropanol solution was determined titrimetrically using 0.01N barium perchlorate as the titrant and thorin as the indicator.

$SO_2$ = The amount absorbed by a 3% hydrogen peroxide solution was determined titrimetrically using 0.01N sodium hydroxide as the titrant and methyl orange as the indicator.

TABLE 8. MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 19.2 (7/31/75) | 19.3 (8/4/75) | 19.4 (8/20/75) | 19.5 (8/22/75) |
|---|---|---|---|---|
| Run Length, hrs. | 10.5 | 6 | 10 | 6 |
| Pressure, kPa | 930 | 930 | 920–930 | 930–940 |
| Lower Bed Temperature, °C | 889 | 897 | 889 | 901 |
| Avg. Bed Temperature, °C | 862–885 | 865–878 | 870–885 | 885–900 |
| Superficial Velocity, m/sec | 1.9 | 1.9 | 1.98–2.0 | 2 |
| Settled Bed Height, m | | | | |
| Initial | 0.71 | 1.45 | 1.58 | 1.98 |
| Final | 1.45 | 1.58 | 1.98 | 1.75 |
| Expanded Bed Height, m | | | | |
| Coal Feed Rate, kg/hr | 93–130 | 136–140 | 132–144 | 126–144 |
| Ca/S Molar Feed Ratio–Set | 1.45 | 1.45 | 2.5 | 2.5 |
| Ca/S Molar Feed Ratio–Calculated | — | 2.40 | 2.65 | 2.60 |
| Excess Air, % | 4.4–8.9 | 5–7 | ~15 | ~15 |
| Sorbent | GL | GL | GL | GL |
| Coal | CH | CH | CH | CH |
| **Flue Gas Emission:** | | | | |
| $SO_2$, ppm | 160–615 | 715–780 | 520 | 537 |
| $NO_x$, ppm | 208–87.5 | 78 | 125 | 91 |
| $CO$, ppm | 50–150 | — | 75–100 | 50 |
| $CO_2$, % | 7.2–10.5 | 12.75 | 12–13 | 11.6–12 |
| $O_2$, % | 6.4–2.7 | 1–1.4 | 2.5–3.0 | 2.7–3.1 |
| **Results:** | | | | |
| $SO_2$ Retention, % | — | 56.4 | 60 | 67 |
| $Ca^{++}$ Sulfation, % | 20.4 | 22.7 | 26 | 25 |
| lb $SO_2$/M BTU | — | 1.26 | 0.92 | 0.87 |
| lb $NO_x$/M BTU | — | 0.10 | 0.16 | 0.12 |

---

GL = Grove Limestone (BCR No. 1359)
CH = Champion Coal

TABLE 8 (Continued). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 19.6 Test 1 (8/26/75) | 19.6 Test 2 (8/26/75) | 19.7 Test 1 (8/28/75) | 19.7 Test 2 (8/28/75) | 19.8 Test 1 (9/11/75) | 19.8 Test 2 (9/11/75) |
|---|---|---|---|---|---|---|
| Run Length, hrs. | 7.5 | 6 | 3.5 | 6 | 6.5 | 6 |
| Pressure, kPa | 930 | 930 | 930 | 970 | 930 | 930 |
| Lower Bed Temperature, °C | — | — | 892 | 967 | 894 | 943 |
| Avg. Bed Temperature, °C | 880–888 | 820–830 | 877–882 | 935–940 | 870 | 926 |
| Superficial Velocity, m/sec | 2.01–2.04 | 1.93–1.95 | 1.98 | 2.16–2.20 | 1.60–1.9 | 2.2–2.26 |
| Settled Bed Height, m | | | | | | |
| Initial | 1.58 | — | 1.96 | — | 1.09 | — |
| Final | — | 1.60 | — | 1.27 | — | 1.00 |
| Expanded Bed Height, m | | | | | | |
| Coal Feed Rate, kg/hr | 131–143 | 120–131 | 131–139 | 139–147 | 106–125 | 131 |
| Ca/S Molar Feed Ratio-Set | 2.5 | 2.5 | 2.5 | 2.5 | 2.5 | 2.5 |
| Ca/S Molar Feed Ratio-Calculated | 3.2 | 3.2 | 4 | 4 | 2.56 | 2.56 |
| Excess Air, % | 15 | 22 | 9.1 | 8.6 | 18 | 20 |
| Sorbent | GL | GL | GL | GL | GL | GL |
| Coal | CH | CH | CH | CH | CH | CH |
| **Flue Gas Emission:** | | | | | | |
| $SO_2$, ppm | 520 | 620 | 450 | 180–340 | 380 | 300 |
| $NO_x$, ppm | 104 | 117 | 102.5 | 141 | 120–150 | 90–120 |
| CO, ppm | — | — | 63 | 132 | 100 | 100 |
| $CO_2$, % | 11.7–12.3 | 11.1 | 10.2 | 15.2 | 14.5–15.2 | 13.6–14 |
| $O_2$, % | 2.5–3.0 | 3.5–4 | 1.7 | 1.8 | 2 | 4 |
| **Results:** | | | | | | |
| $SO_2$ Retention, % | 68 | 58 | 73 | 83.5 | 66 | 81.9 |
| Ca Sulfation, % | 21 | 18 | — | 20.9 | — | 32 |
| Lb $SO_2$/M BTU | 0.95 | 1.23 | 0.85 | — | 1.31 | 0.65 |
| Lb $NO_x$/M BTU | 0.14 | 0.81 | 0.14 | 0.19 | 0.20 | 0.16 |

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GL = Grove Limestone (BCR No. 1359)
CH = Champion Coal

TABLE 9 (Continued). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 20.1 Test 1 (9/17) | 20.1 Test 2 (9/17) | 20.1 Test 3 (9/17) | 20.2 Test 1 (9/22) | 20.2 Test 2 (9/22) |
|---|---|---|---|---|---|
| Run Length, hrs. | 6 | 2 | 2.5 | 6 | 3.3 |
| Pressure, kPa | 930 | 930 | 930 | 930 | 930 |
| Lower Bed Temperature, °C | — | — | — | 967 | 967 |
| Avg. Bed Temperature, °C | 870 | 827 | 927 | 927 | 927 |
| Superficial Velocity, m/sec | 2.06 | 1.83 | 2.32 | 2.3 | 2.3 |
| Settled Bed Height, m | | | | | |
| Initial | 1.85 | — | — | 1.22 | — |
| Final | — | — | 1.32 | — | 1.65 |
| Expanded Bed Height, m | | | | | |
| Coal Feed Rate, kg/hr | 127 | 116 | 136 | 127 | 127 |
| Ca/S Molar Feed Ratio-Set | 3.3 | 3.3 | 3.3 | 1.50 | 3.3 |
| Ca/S Molar Feed Ratio-Calculated | — | — | — | — | 3.75 |
| Excess Air, % | 11 | 12 | 18.5 | 18 | 13.7 |
| Sorbent | GL | GL | GL | GL | GL |
| Coal | CH | CH | CH | CH | CH |
| **Flue Gas Emission:** | | | | | |
| $SO_2$, ppm | 720 | 725 | 200-250 | 420 | 200 |
| $NO_x$, ppm | 90-145 | 95 | 130-185 | 208 | 195 |
| CO, ppm | ~100 | ~100 | ~100 | 130 | 130 |
| $CO_2$, % | 14.5-15.1 | — | — | 14.5-14.75 | 14.5-14.75 |
| $O_2$, % | 2.1 | 2.25 | 3.0 | 2.5 | 2.5 |
| **Results:** | | | | | |
| $SO_2$ Retention, % | 56 | 53 | — | 71.2 | 88.3 |
| Ca Sulfation, % | — | — | — | — | 23 |
| Lb $SO_2$/MM BTU | 1.45 | 1.5 | — | 1.26 | 0.71 |
| Lb $NO_x$/MM BTU | 0.17 | 0.14 | — | 0.33 | 0.32 |

GL = Grove Limestone (BCR No. 1359)
CH - Champion Coal

TABLE 8 (Continued). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 21 Test 1 (9/22) | 21 Test 2 (9/22) | 22 (10/2) | 23 (10/3) | 25 (10/9) | 26 Test 1 (10/24) | 26 Test 2 (10/24) |
|---|---|---|---|---|---|---|---|
| Run Length, hrs. | 8.5 | 4.5 | 10 | 7 | 5.5 | 11.25 | 6.25 |
| Pressure, kPa | 930 | 930 | 930 | 930 | 930 | 930 | 930 |
| Lower Bed Temperature, °C | | | | | 850 | 948 | — |
| Avg. Bed Temperature, °C | 870 | 930 | 870 | 885 | 970 | 927 | 885 |
| Superficial Velocity, m/sec | 2.0 | 2.2 | 1.90 | 1.72-1.92 | 1.75 | 2.1 | 1.90 |
| Settled Bed Height, m | | | | | | | |
| Initial | 1.6 | — | 2.11 | 1.19 | 1.37 | 1.12 | — |
| Final | — | 2.11 | 1.19 | 1.05 | 1.78 | — | 2.28 |
| Expanded Bed Height, m | | | | | | | |
| Coal Feed Rate, kg/hr | 127-130 | 127-130 | 113 | ~120 | ~120 | 130 | 130 |
| Ca/S Molar Feed Ratio-Set | 3.3 | 3.3 | 0 | 1.3 | 1.3 | 3.7 | 3.7 |
| Ca/S Molar Feed Ratio-Calculated | — | — | — | — | — | 2.75 | 2.75 |
| Excess Air, % | 14 | 18 | 28 | 12 | 3 | 9.5 | 11.5 |
| Sorbent | GL | GL | GL | TD | PD | GL | GL |
| Coal | CH | CH | CH | CH | CH | CH | CH |
| **Flue Gas Emissions:** | | | | | | | |
| $SO_2$, ppm | 600 | 0-230 | 600-1040 | 810-270 | 245 | 240 | 300 |
| $NO_x$, ppm | 195 | 210 | — | 100-115 | 125 | 185 | 180 |
| CO, ppm | 100 | 100 | 30 | 100 | 200 | 130 | 130 |
| $CO_2$, % | 14.2 | 16.3 | 13 | 12.75 | 14.5 | 12.75 | 13 |
| $O_2$, % | 2.5 | 3.2 | 4.25-4.75 | 2.25 | 1.0-1.75 | 1.8 | 2.15 |
| **Results:** | | | | | | | |
| $SO_2$ Retention, % | 60 | — | — | — | — | 90.8 | 81.2 |
| Ca Sulfation, % | — | — | — | — | — | 33 | 29 |
| lb $SO_2$/M BTU | 1.18 | — | — | — | — | 0.28 | 0.56 |
| lb $NO_x$/M BTU | 0.26 | 0.31 | — | 0.15 | 0.17 | 0.26 | 0.24 |

GL = Grove Limestone (BCR No. 1359)
TD = Tymochtee Dolomite
PD = Pfizer Dolomite (BCR No. 1337)
CH = Champion Coal

TABLE 8 (Continued). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 27.1 (10/27-28) | 27.2 (10/28) | 27.3 (10/28-29) | 27.4 (10/29) |
|---|---|---|---|---|
| Run Length, hrs | 13.5 | 14 | 10 | 13 |
| Pressure, kPa | 930 | 930 | 930 | 930 |
| Lower Bed Temperature, °C | 902 | 956 | 902 | 843 |
| Avg. Bed Temperature, °C | 880 | 927 | 881 | 829 |
| Superficial Velocity, m/sec | 1.01 | 1.15 | 1.00 | 1.07 |
| Settled Bed Height, m | | | | |
| Initial | — | — | — | — |
| Final | — | — | — | — |
| Expanded Bed Height, m | ~4 | ~4 | ~3 | — |
| Coal Feed Rate, kg/hr | 135 | 139 | 133 | 123 |
| Ca/S Molar Feed Ratio-Set | 0.5 | 0.5 | 0.5 | 0.5 |
| Ca/S Molar Feed Ratio-Calculated | 0.8 | 0.8 | 0.84 | 0.91 |
| Excess Air, % | 18.9 | 15.4 | 15.3 | 20.5 |
| Sorbent | PD | PD | PD | PD |
| Coal | CH | CH | CH | CH |
| **Flue Gas Emission:** | | | | |
| $SO_2$, ppm | 659 | 612 | 630 | 532 |
| $NO_x$, ppm | 207 | 194 | 176 | 208 |
| CO, ppm | 83 | 88 | 93 | 46 |
| $CO_2$, % | 14.5 | 15.8 | 11 | 16.1 |
| $O_2$, % | 3.3 | 2.7 | 2.8 | 3.6 |
| **Results:** | | | | |
| $SO_2$ Retention, % | 55 | 57.1 | 55.6 | 62 |
| Ca Sulfation, % | 66 | 70 | 66 | 68 |
| lb $SO_2$/M BTU | 1.17 | 1.12 | 1.16 | 1.05 |
| lb $NO_2$/M BTU | 0.27 | 0.27 | 0.24 | 0.28 |

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PD = Pfizer Dolomite (BCR No. 1337)
CH = Champion Coal

## TABLE 8 (Continued). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 27.5 (10/29-30) | 27.6 (10/30-31) | 27.7 (10/31) | 27.8 (10/31) |
|---|---|---|---|---|
| Run Length, hrs. | 27 | 8.5 | 11 | 7 |
| Pressure, kPa | 930 | 930 | 930 | 930 |
| Lower Bed Temperature, °C | 844 | 842 | 847 | 898 |
| Avg. Bed Temperature, °C | 829 | 829 | 835 | 878 |
| Superficial Velocity, m/sec | 1.71 | 1.72 | 1.72 | 1.83 |
| Settled Bed Height, m | | | | |
| Initial | — | — | — | — |
| Final | — | — | — | — |
| Expanded Bed Height, m | ~6 | ~5 | ~6 | ~7 |
| Coal Feed Rate, kg/hr | 122 | 123 | 132 | 134 |
| Ca/S Molar Feed Ratio-Set | 2.5 | 0.8 | 1.5 | 1.5 |
| Ca/S Molar Feed Ratio-Calculated | 2.2 | — | 1.85 | 1.85 |
| Excess Air, % | 12.1 | 14.5 | 12.4 | 12.6 |
| Sorbent | PD | PD | PD | PD |
| Coal | CH | CH | CH | CH |
| **Flue Gas Emission:** | | | | |
| $SO_2$, ppm | 34 | 306 | 47 | 28 |
| $NO_x$, ppm | 189 | 143 | 135 | 128 |
| CO, ppm | 81 | 77 | 106 | 81 |
| $CO_2$, % | 13.8 | 16.3 | 16.6 | 16.6 |
| $O_2$, % | 2.3 | 1.6 | 2.3 | 2.3 |
| **Results:** | | | | |
| $SO_2$ Retention, % | 97.8 | 80 | 97.2 | 98 |
| Ca Sulfation, % | 44 | — | 53 | 53 |
| lb $SO_2$/M BTU | 0.06 | 0.56 | 0.08 | 0.05 |
| lb $NO_x$/M BTU | 0.27 | 0.19 | 0.17 | 0.16 |

PD = Pfizer Dolomite (BCR No. 1337)
CH = Champion Coal

TABLE 9 (Continued). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 27.9 (10/31) | 27.10 (11/1) | 27.11 (11/1-2) | 27.12 (11/2) | 27.13 (11/2) |
|---|---|---|---|---|---|
| Run Length, hrs. | 3 | 17 | 11 | 10.5 | 8 |
| Pressure, kPa | 930 | 930 | 930 | 930 | 930 |
| Lower Bed Temperature, °C | 948 | 900 | 952 | 842 | 890 |
| Avg. Bed Temperature, °C | 922 | 883 | 931 | 834 | 888 |
| Superficial Velocity, m/sec | 2.09 | 1.87 | 2.23 | 1.96 | 2.02 |
| Settled Bed Height, m | | | | | |
| Initial | — | — | — | — | — |
| Final | — | — | — | — | — |
| Expanded Bed Height, m | ~5 | ~5 | ~7 | ~7 | ~6 |
| Coal Feed Rate, kg/hr | 142 | 135 | 149 | 133 | 136 |
| Ca/S Molar Feed Ratio-Set | 1.5 | 0.75 | 0.75 | 0.75 | 0.35 |
| Ca/S Molar Feed Ratio-Calculated | 2.1 | 1.03 | 1.0 | 1.03 | 0.6 |
| Excess Air, % | 13.1 | 7.8 | 11 | 23 | 13.3 |
| Sorbent | PD | PD | PD | PD | PD |
| Coal | CH | CH | CH | CH | CH |
| **Flue Gas Emission:** | | | | | |
| $SO_2$, ppm | 24 | 455 | 444 | 488 | 822 |
| $NO_x$, ppm | 194 | 93 | 109 | 130 | 110 |
| CO, ppm | 75 | 109 | 71 | 76 | 66 |
| $CO_2$, % | 17.4 | 15.1 | 16.4 | 15.1 | 16 |
| $O_2$, % | 2.4 | 1.5 | 2 | 3.9 | 2.5 |
| **Results:** | | | | | |
| $SO_2$ Retention, % | 98.5 | 72 | 70 | 71 | 46 |
| Ca Sulfation, % | 47 | 70 | 70 | 70 | 78 |
| Lb $SO_2$/MM BTU | 0.04 | 0.79 | 0.79 | 0.76 | 1.34 |
| Lb $NO_x$/MM BTU | 0.21 | 0.11 | 0.14 | 0.17 | 0.15 |

PD = Pfizer Dolomite (BCR No. 1337)
CH = Champion Coal

TABLE 8 (Continued). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 27.14 (11/2-3) | 27.15 (11/3-4) | 27.16 (11/4) | 27.17 (11/4) | 27.18 (11/5) |
|---|---|---|---|---|---|
| Run Length, hrs. | 11 | 24.5 | 6.5 | 8 | 5 |
| Pressure, kPa | 930 | 930 | 930 | 930 | 937 |
| Lower Bed Temperature, °C | 898 | 896 | 844 | 889 | 919 |
| Avg. Bed Temperature, °C | 892 | 889 | 842 | 891 | 909 |
| Superficial Velocity, m/sec | 1.04 | 1.48 | 1.74 | 1.87 | 1.89 |
| Settled Bed Height, m | | | | | |
| Initial | — | — | — | — | — |
| Final | — | — | — | — | — |
| Expanded Bed Height, m | ~6 | ~5 | ~5 | ~6 | ~7 |
| Coal Feed Rate, kg/hr | 137 | 136 | 129 | 130 | 143 |
| Ca/S Molar Feed Ratio-Set | 0.73 | 1 | 0.75 | 0.75 | 1 |
| Ca/S Molar Feed Ratio-Calculated | 0.7 | 0.97 | 1.12 | 1.12 | — |
| Excess Air, % | 14.6 | 8.2 | 10.4 | 13.8 | 8.2 |
| Sorbent | PD | PD | PD | PD | PD |
| Coal | CB | CB | CB | CB | CB |
| **Flue Gas Emission:** | | | | | |
| $SO_2$, ppm | 368 | 465 | 206 | 456 | 269 |
| $NO_x$, ppm | 100 | 103 | 96 | 85 | 97 |
| CO, ppm | 70 | 107 | 29 | 36 | 37 |
| $CO_2$, % | 16 | 14.7 | 11 | 14 | 16.8 |
| $O_2$, % | 2.7 | 1.6 | 2 | 2.5 | 1.7 |
| **Results:** | | | | | |
| $SO_2$ Retention, % | 84 | 71 | 87 | 72 | 86.5 |
| Ca Sulfation, % | 92 | 73 | — | 64 | — |
| lb $SO_2$/M BTU | 1.05 | 0.86 | 0.36 | 0.81 | 0.43 |
| lb $NO_x$/M BTU | 0.13 | 0.13 | 0.12 | 0.12 | 0.12 |

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PD = Pfizer Dolomite (BCR No. 1337)

CB = Champion Coal

TABLE 9 (Continued). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 27.19 (11/5) | 27.20 (11/5) | 27.21 (11/6) | 28.1 (12/15) | 28.2 (12/15) |
|---|---|---|---|---|---|
| Run Length, hrs. | 7 | 10.5 | 13.5 | 4.25 | 2.5 |
| Pressure, kPa | 930 | 930 | 930 | 930 | 930 |
| Lower Bed Temperature, °C | 915 | 897 | 844 | — | — |
| Avg. Bed Temperature, °C | 927 | 885 | 846 | 840 | 870 |
| Superficial Velocity, m/sec | 2.09 | 2.03 | 1.67 | 2.13 | 2.16 |
| Settled Bed Height, m | | | | | |
| Initial | — | — | — | 1.02 | — |
| Final | — | — | — | — | — |
| Expanded Bed Height, m | ~7 | ~7 | ~5 | | |
| Coal Feed Rate, kg/hr | 137 | 138 | 125 | 105 | 109 |
| Ca/S Molar Feed Ratio-Set | 1.5 | 0 | 0 | 0 | 0 |
| Ca/S Molar Feed Ratio-Calculated | 1.5 | — | — | — | — |
| Excess Air, % | 11.3 | 11.1 | 10.9 | 40 | 35 |
| Sorbent | PD | PD | PD | Alumina | Alumina |
| Coal | CH | CH | CH | CH | CH |
| **Flue Gas Emissions:** | | | | | |
| $SO_2$, ppm | 150 | 1171 | 1290 | 975 | 1013 |
| $NO_x$, ppm | 101 | 91 | 65 | 136 | 145 |
| CO, ppm | 45 | 34 | 56 | 45 | 45 |
| $CO_2$, % | 15.3 | 11.9 | 15.8 | 10 | 11 |
| $O_2$, % | 2.3 | 2.3 | 2 | 6.2 | 5.4 |
| **Results:** | | | | | |
| $SO_2$ Retention, % | 90 | 11 | 24 | 10 | 13.5 |
| Ca Sulfation, % | — | — | — | — | — |
| Lb $SO_2$/M BTU | 0.29 | 2.33 | 2.24 | 2.7 | 2.68 |
| Lb $NO_x$/M BTU | 0.13 | 0.12 | 0.08 | 0.27 | 0.27 |

PD = Pfizer Dolomite (BCR No. 1337)
CH = Champion Coal

TABLE 9 (Continued). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 28.3 (12/25) | 28.4 (12/26) | 28.5 (12/26/75) | 29 (1/15/76) | 34.1 (1/27/76) |
|---|---|---|---|---|---|
| Run Length, hrs. | 2.5 | 2 | 2.5 | 7.3 | 7 |
| Pressure, kPa | 930 | 930 | 930 | 930 | 920 |
| Lower Bed Temperature, °C | — | — | — | 887 | 889 |
| Avg. Bed Temperature, °C | 920 | 920 | 930 | 875 | 885 |
| Superficial Velocity, m/sec | 2.13 | 1.40 | 2.65 | 2.26 | 2.15 |
| Settled Bed Height, m | | | | | |
| Initial | — | — | [illegible] | 0.8 | 1.6 |
| Final | — | — | [illegible] | 1.6 | — |
| Expanded Bed Height, m | | | | | |
| Coal Feed Rate, kg/hr | 114 | 91 | 123 | 138 | 133 |
| Ca/S Molar Feed Ratio-Set | 0 | 0 | 0 | 3.7 | 3.7 |
| Ca/S Molar Feed Ratio-Calculated | — | — | — | 2.8 | 1.5 |
| Excess Air, % | 18 | 4.5 | 21 | 19 | 13.7 |
| Sorbent | Alumina | Alumina | Alumina | GL | GL |
| Coal | CH | CH | CH | CH | CH |
| **Flue Gas Emission:** | | | | | |
| $SO_2$, ppm | 1163 | 1575 | 1088 | 623 | 750 |
| $NO_x$, ppm | 125 | 41 | 146 | 137 | 50 |
| CO, ppm | 45 | 11 | 11 | 167 | 175-200 |
| $CO_2$, % | 11 | 16 | 12 | 14.6 | 14.9 |
| $O_2$, % | 3.1 | 0.9 | 3.6 | 3.4 | 2.5 |
| **Results:** | | | | | |
| $SO_2$ Retention, % | 9 | 1.6 | 3.6 | 55.7 | 48.7 |
| Ca Sulfation, % | — | — | — | 19.9 | 32.9 |
| lb $SO_2$/M BTU | 2.76 | 3.08 | 2.95 | 1.28 | 1.57 |
| lb $NO_2$/M BTU | 0.22 | 0.76 | 0.26 | 0.2 | 0.08 |

GL = Grove Limestone (OGR No. 1359)
CH = Champion Coal

TABLE 8 (Continued). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 30.2 (1/27-28) | 30.3 (1/28) | 30.4 (1/28) | 31 (2/4-5) | 32.1 (2/10) |
|---|---|---|---|---|---|
| Run Length, hrs. | 8.5 | 10 | 6 | 12 | 8.5 |
| Pressure, kPa | 920 | 920 | 920 | 520 | 600 |
| Lower Bed Temperature, °C | 945 | 886 | 835 | — | — |
| Avg. Bed Temperature, °C | 929 | 885 | 825 | 830 | 950 |
| Superficial Velocity, m/sec | 2.5 | 2.1 | 1.9 | 1.96 | 2.2 |
| Settled Bed Height, m | | | | | |
| Initial | — | — | — | 1.6 | 1.4 |
| Final | — | — | 2.26 | 1.35 | — |
| Expanded Bed Height, m | | | | | |
| Coal Feed Rate, kg/hr | 137 | 130 | 120 | 82 | 87 |
| Ca/S Molar Feed Ratio-Set | 3.7 | 3.7 | 3.7 | 2.5 | 2.5 |
| Ca/S Molar Feed Ratio-Calculated | 3.3 | 1.65 | 1.1 | 3 | 3.2 |
| Excess Air, % | 17.2 | 13.9 | 26.1 | 23 | 13 |
| Sorbent | GL | GL | GL | GL | GL |
| Coal | CH | CH | CH | CH | CH |
| **Flue Gas Emission:** | | | | | |
| $SO_2$, ppm | 137 | 480 | 894 | 525 | 330 |
| $NO_x$, ppm | — | — | 70 | 55 | 80 |
| CO, ppm | — | — | — | 200 | 175 |
| $CO_2$, % | 15.1 | 15.3 | 14.4 | 12.7 | 13.5 |
| $O_2$, % | 3.1 | 2.5 | 2.8 | 4.4 | 2.5 |
| **Results:** | | | | | |
| $SO_2$ Retention, % | 89.1 | 64.5 | 32.1 | 66 | 76.4 |
| Ca Sulfation, % | 27 | 39.1 | 30 | 22 | 24 |
| Lb $SO_2$/MM BTU | 0.31 | 1.0 | 1.92 | 0.96 | 0.67 |
| Lb $NO_x$/MM BTU | — | — | 0.10 | 0.07 | 0.12 |

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GL = Grove Limestone (BCR No. 1359)
CH = Champion Coal

TABLE 9 (Continued). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 32.2 (2/10-11) | 32.3 (2/11) | 33 (3/8) | 34 (3/11) | 35 (3/17) |
|---|---|---|---|---|---|
| Run Length, hrs. | 11.3 | 8.25 | 14 | 13.25 | 6.5 |
| Pressure, kPa | 600 | 600 | 920 | 932 | 930 |
| Lower Bed Temperature, °C | — | — | 880 | 868 | 904 |
| Avg. Bed Temperature, °C | 947 | 836 | 919 | 900 | 905 |
| Superficial Velocity, m/sec | 1.1 | 1.5 | 1.56 | 1.5 | 1.56 |
| Settled Bed Height, m | | | | | |
| Initial | — | — | 0.76 | 1.29 | 1.28 |
| Final | — | 0.9 | 1.29 | 1.29 | 1.28 |
| Expanded Bed Height, m | | | 4.6 | 4.6 | 4.6 |
| Coal Feed Rate, kg/hr | 89 | 68 | 95 | 90 | 80 |
| Ca/S Molar Feed Ratio-Set | 0.75 | 0.75 | 1.5 | 0.75 | 0.75 |
| Ca/S Molar Feed Ratio-Calculated | 1.5 | 1.5 | — | 0.9 | 0.9 |
| Excess Air, % | 16 | 20 | 18.1-28.7 | 20.9 | 45.8 |
| Sorbent | PD | PD | PD | PD | PD |
| Coal | CH | CH | CH | CH | CH |
| **Flue Gas Emission:** | | | | | |
| $SO_2$, ppm | 600 | 490 | ~0 | *120-300 | 442 |
| $NO_x$, ppm | 106 | 89 | 65-130 | 52 | 72 |
| CO, ppm | 200 | 200 | 300 | 200 | 225 |
| $CO_2$, % | 14.3 | 14.3 | 13.1-15 | 15.5 | 13 |
| $O_2$, % | 1.9 | 1.6 | 3.2-6.6 | 3.5 | 6.5 |
| **Results:** | | | | | |
| $SO_2$ Retention, % | 60 | 66 | — | — | 82.6 |
| Ca Sulfation, % | 40 | 44 | — | — | 69 |
| lb $SO_2$/M BTU | 1.16 | 0.96 | — | — | 1.1 |
| lb $NO_x$/M BTU | 1.44 | 0.12 | — | — | 0.13 |

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CH = Champion Coal

PD = Pfizer Dolomite (BCR no. 1337)

*Water on Sampling Line Affected Readings.

TABLE 9 (Continued). MINIPLANT FLUIDIZED-BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 36.1 (3/24) | 36.2 (3/24) | 37 (4/7) | 38.1 (4/24) | 38.2 (4/24) |
|---|---|---|---|---|---|
| Run Length, hr. | 6.5 | 5 | 13.5 | 7.3 | 6 |
| Pressure, kPa | 930 | 930 | 930 | 930 | 930 |
| Lower Bed Temperature, °C | 906 | 932 | 899 | 888 | 889 |
| Avg. Bed Temperature, °C | 900 | 913 | 902 | 891 | 894 |
| Superficial Velocity, m/sec | 2.6 | 3 | 2.96 | 2.08 | 2.09 |
| Settled Bed Height, m | | | | | |
| Initial | 1.4 | — | 1.68 | 1.14 | — |
| Final | — | 1.7 | 2.17 | — | — |
| Expanded Bed Height, m | 2.6 | 3 | 4 | 2.9 | 3.6 |
| Coal Feed Rate, kg/hr | 80 | 120 | 150 | 90 | 90 |
| Ca/S Molar Feed Ratio-Set | 0.75 | 0.75 | 0.75 | 0.75 | 0.75 |
| Ca/S Molar Feed Ratio-Calculated | 1.3 | 1.3 | 0.5 | 0.42 | 0.43 |
| Excess Air, % | 42 | 96 | 92 | 47 (1) | 44 (2) |
| Sorbent | PD | PD | PD | PD | PD |
| Coal | CH | CH | CH | CH | CH |
| **Flue Gas Emissions:** | | | | | |
| $SO_2$, ppm | 380 | 202 | 447 | 762 | 690 |
| $NO_x$, ppm | 94 | 159 | 121 | 135 | 127 |
| CO, ppm | 200 | 180 | 50 | 175 | 225 |
| $CO_2$, % | 11.8 | 8.8 | 9.75 | 8.5 | 8 |
| $O_2$, % | 6 | 10 | 10 | 6.6 | 6.3 |
| **Results:** | | | | | |
| $SO_2$ Retention, % | 65.6 | 75.6 | 46 | 40 | 45 |
| Ca Sulfation, % | — | 58 | 90 | 66 | 72 |
| lb $SO_2$/MM BTU | 0.98 | 0.71 | 1.53 | 2.29 | 2.08 |
| lb $NO_x$/MM BTU | 0.17 | 0.39 | 0.3 | 0.29 | 0.27 |

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CH = Champion Coal

PD = Pfizer Dolomite (No. 1337)

(1) 14 X 25 Mesh Sorbent Particle Size

(2) 8 X 14 Mesh Sorbent Particle Size

TABLE 8 (Continued). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 38.3 (4/14-15) | 38.4 (4/15) | 38.5 (4/15) | 38.6 (4/15) | 39.1 (5/29) |
|---|---|---|---|---|---|
| Run Length, hrs. | 6 | 8.25 | 1.5 | 2 | 11 |
| Pressure, kPa | 930 | 930 | 930 | 930 | 902 |
| Lower Bed Temperature, °C | 758 | 758 | 681 | 678 | 756 |
| Avg. Bed Temperature, °C | 762 | 762 | 690 | 684 | 750 |
| Superficial Velocity, m/sec | 1.85 | 1.84 | 1.7 | 1.3 | 1.52 |
| Settled Bed Height, m | | | | | |
| Initial | — | — | — | — | 1.22 |
| Final | — | — | — | 2.26 | — |
| Expanded Bed Height, m | 3.76 | 4.75 | 4.75 | 4.56 | 1.25 |
| Coal Feed Rate, kg/hr | 76 | 78 | 75 | 67 | 74 |
| Ca/S Molar Feed Ratio-Set | 0.75 | 1.5 | 1.5 | 1.5 | 2.5 |
| Ca/S Molar Feed Ratio-Calculated | 0.94 | 1.14 | 1.49 | 1.43 | — |
| Excess Air, % | 94 | 94 | 112 | 78 | 60 |
| Sorbent | PD | PD | PD | PD | GL |
| Coal | CH | CH | CH | CH | CH |
| **Flue Gas Emission:** | | | | | |
| $SO_2$, ppm | 388 | 223 | 180 | 163 | 912 |
| $NO_x$, ppm | 125 | 169 | 114 | 102 | 120 |
| CO, ppm | 325 | 375 | 800 | 400 | 350 |
| $CO_2$, % | 6.47 | 6.6 | 5.91 | 7.08 | 9.56 |
| $O_2$, % | 7.48 | 10 | 10.9 | 9.1 | 8.37 |
| **Results:** | | | | | |
| $SO_2$ Retention, % | 48 | 71 | 76 | 82 | 0 |
| Ca Sulfation, % | 51 | 62 | 51 | 57 | — |
| lb $SO_2$/M BTU | 1.42 | 0.78 | 0.64 | 0.5 | 2.69 |
| lb $NO_x$/M BTU | 0.32 | 0.37 | 0.29 | 0.23 | 0.26 |

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CH = Champion Coal

PD = Pfizer Dolomite (BCR No. 1337)

GL = Grove Limestone (BCR No. 1359)

TABLE 8 (Continued). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

| Operating Conditions: | 39.2 (5/19-20) | 39.3 (5/20) | 39.4 (5/20-21) |
|---|---|---|---|
| Run Length, hrs. | 6.6 | 15 | 5 |
| Pressure, kPa | 902 | 932 | 932 |
| Lower Bed Temperature, °C | 673 | 946 | 944 |
| Avg. Bed Temperature, °C | 674 | 938 | 938 |
| Superficial Velocity, m/sec | 1.4 | 1.56 | 1.58 |
| Settled Bed Height, m | | | |
| Initial | — | — | — |
| Final | — | — | 1.88 |
| Expanded Bed Height, m | 2.4 | 3.55 | 3.76 |
| Coal Feed Rate, kg/hr | 73 | 80 | 81 |
| Ca/S Molar Feed Ratio-Set | 2.5 | 2.5 | 2.5 |
| Ca/S Molar Feed Ratio-Calculated | — | 3.5 | 3.8 |
| Excess Air, % | 60 | 32 (1) | 30 (2) |
| Sorbent | GL | GL | GL |
| Coal | CH | CH | CH |
| **Flue Gas Emission:** | | | |
| $SO_2$, ppm | 900 | 329 | 345 |
| $NO_x$, ppm | 176 | 125 | 115 |
| CO, ppm | — | 108 | 100 |
| $CO_2$, % | 9.37 | 12.86 | 11.93 |
| $O_2$, % | 7.7 | 4.9 | 4.88 |
| **Results:** | | | |
| $SO_2$ Retention, % | 0.6 | 70.3 | 68.7 |
| Ca Sulfation, % | — | — | 20 |
| lb $SO_2/10^6$ BTU | 2.66 | 0.80 | 0.84 |
| lb $NO_2/10^6$ BTU | 0.37 | 0.22 | 0.2 |

CH = Champion Coal
GL = Grove Limestone (BCR No. 1359)
(1) 8 X 14 Mesh Sorbent Particle Size
(2) 14 X 25 Mesh Sorbent Particial Size

TABLE C. DETERMINATION OF $SO_2$ AND $SO_3$ BY WET CHEMISTRY

| Run No. | Sampling[(1)] Location | Wet Chemistry Analysis $SO_2$ (ppm) | Wet Chemistry Analysis $SO_3$ (ppm) | U.V. Monitored Value $SO_2$ (ppm) | Ratio of Wet Chemistry and U.V. Analysis for $SO_2$ (x100) |
|---|---|---|---|---|---|
| 19.2 | Third Deck | 130 | 24 | 180 | 77 |
| 19.2 | ↓ | 445 | 95 | 505 | 88 |
| 19.2 | | 679 | 64 | 732 | 93 |
| 19.3 | | 692 | 11 | 750 | 92 |
| 19.4 | | 257 | 2 | 500 | 51 |
| 19.4 | | 445 | 6 | 500 | 89 |
| 19.5 | | 608 | 164 | 500 | 121 |
| 19.6 | | 445 | 5 | 500 | 89 |
| 19.6 | | 475 | 10 | 500 | 95 |
| 19.7 | | 346 | 6 | 450 | 77 |
| 19.7 | | 173 | 18 | 200 | 87 |
| 19.8 | | 751 | 9 | 686 | 110 |
| 20.1 | | 712 | 4 | 730 | 98 |
| 21 | | 346 | 4 | 460 | 75 |
| 21 | | 425 | 6 | 460 | 90 |
| 22 | | 0 | 0 | 0 | 100 |
| 22 | | 50 | 12 | 105 | 48 |
| 22 | | 356 | 0 | 250 | 142 |
| 22 | | 485 | 262 | 1075 | 50 |
| 22 | | 850 | 5 | 1040 | 82 |
| 23 | | 326 | 1 | 305 | 107 |
| 26 | | 188 | 0 | 250 | 75 |
| 26 | | 198 | 0 | 162 | 122 |
| 26 | | 59 | 0 | 140 | 42 |
| 26 | | 128 | 0 | 220 | 59 |
| 26 | | 208 | 0 | 300 | 69 |
| 27.2 | | 340 | 59 | 390 | 87 |
| 27.2 | | 470 | 94 | 660 | 71 |
| 27.3 | | 550 | 31 | 630 | 87 |
| 27.3 | | | | | |
| 27.4 | | 390 | 19 | 450 | 87 |
| 27.4 | | 450 | 18 | 510 | 88 |
| 27.5 | | 70 | 2 | 75 | 93 |
| 27.5 | | 40 | 0 | 30 | 133 |

TABLE C (Continued). DETERMINATION OF $SO_2$ AND $SO_3$ BY WET CHEMISTRY

| Run No. | Sampling[1] Location | Wet Chemistry Analysis $SO_2$ (ppm) | Wet Chemistry Analysis $SO_3$ (ppm) | U.V. Monitored Value $SO_2$ (ppm) | Ratio of Wet Chemistry and U.V. Analyses for $SO_2$ (x100) |
|---|---|---|---|---|---|
| 27.6 | Third Deck | 50 | 8 | 240 | 21 |
| 27.7 | | 20 | 7 | 39 | 51 |
| 27.10 | | 780 | 52 | — | — |
| 27.11 | | 0 | 30 | 90 | — |
| 27.11 | | 190 | 7 | 360 | 53 |
| 27.12 | | 210 | 30 | 330 | 64 |
| 27.13 | | 710 | 29 | 720 | 99 |
| 27.14 | | 410 | 30 | 480 | 85 |
| 27.15 | | 350 | — | 450 | 78 |
| 27.16 | | 90 | 16 | 60 | 150 |
| 27.17 | | 320 | 23 | 375 | 85 |
| 27.17 | | 400 | 23 | 420 | 95 |
| 27.19 | | 70 | 12 | 180 | 39 |
| 27.18 | | 790 | 14 | 900 | 88 |
| 27.20 | | 1240 | 35 | 1280 | 97 |
| 27.21 | | 1150 | 45 | 1200 | 96 |
| 34 | Control Room | 297 | 0 | 266 | 112 |
| 34 | Third Deck | 129 | 0 | 266 | 48 |
| 35 | | 119 | — | 349 | 34 |
| 35 | | 150 | 20 | 407 | 37 |
| 38.1 | | 536 | 31 | 755 | 71 |
| 38.1 | Control Room | 623 | 16 | 825 | 76 |
| 38.2 | Third Deck | 227 | 0 | 675 | 34 |
| 38.2 | Control Room | 563 | 0 | 623 | 90 |
| 38.3 | Third Deck | 365 | 0 | 420 | 87 |
| 38.3 | Control Room | 395 | 0 | 400 | 99 |
| 39.1 | Control Room | 880 | 0 | 912 | 96 |
| 39.2 | Control Room | 910 | 0 | 900 | 101 |
| 39.3 | Control Room | 327 | 0 | — | |
| 39.4 | Control Room | 405 | 0 | 450 | 90 |
| Calibration Gas | | 306 | — | 290 | 105 |
| Calibration Gas | | 270 | — | 290 | 93 |
| Calibration Gas | | 1318 | — | 1320 | 100 |
| Calibration Gas | | 1532 | — | 1490 | 103 |

(1) Third Deck - At the flue gas sampling port on the off-gas line prior to the sample preparation system
Control Room - Prior to the continuous analyzers and after sample preparation

## TABLE 11. ENTRAINMENT RATES FOR GROVE NO. 1359 LIMESTONE WITH LIMITED CALCINATION

| Miniplant Run No. | Ca/S (Mole/Mole) | Superficial Velocity (m/sec) | Volume Bed Entrained Per Hr. | Entrainment Losses Ca/S Equiv. (Mole/Mole) | Wt. Percent Input Ca Lost by Entrainment | Remarks |
|---|---|---|---|---|---|---|
| 29.2 | 1.45 | 1.9 | 2.8 | 0.40 | 27 | Based on flyash sample representative of the entire run. |
| 29.3 | 1.45 | 1.9 | 0.9 | 0.18 | 12 | |
| 29.4 | 2.65 | 2.0 | 0.8 | 0.20 | 8 | |
| 29.5 | 2.7 | 2.0 | 2.3 | 0.50 | 19 | |
| 26 | 2.75 | 2.0 | 1.6 | 0.40 | 14 | |
| 28.1 | 2.8 | 2.16 | 1.5 | 0.25 | 9 | Based on flyash samples taken at one-hour intervals |
| 30.1 | 1.5 | 2.15 | 1.1 | 0.21 | 14 | |
| | | | 1.2 | 0.22 | 15 | |
| | | | 1.3 | 0.24 | 16 | |
| 30.3 | 1.6 | 2.1 | 1.0 | 0.21 | 13 | |
| | | | 1.1 | 0.22 | 14 | |
| | | | 1.0 | 0.20 | 13 | |
| 31 | 3.0 | 1.96 | 1.6 | 0.30 | 13 | |
| | | | 1.0 | 0.24 | 8 | |
| 39.1 | 2.5 | 1.52 | 0.5 | 0.15 | 6 | |
| | | | 0.8 | 0.25 | 10 | |
| | | | 0.8 | 0.25 | 10 | |
| 39.2 | 2.5 | 1.4 | 0.4 | 0.13 | 5 | |
| | | | 0.4 | 0.12 | 5 | |
| | | | 0.4 | 0.11 | 4 | |
| | | Avg. | 1.1 ± 0.6 | 0.24 ± 0.1 | 12 ± 7 | |

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Pressure - 930 kPa except Run 31 which was at 520 kPa

Temperature - 815°C - 905°C except Run 39.1 at 760°C and Run 39.2 at 680°C.

## TABLE 6. ENTRAINMENT RATES FOR GROVE NO. 1359 LIMESTONE WITH EXTENSIVE CALCINATION

| Miniplant Run No. | (Mole/Mole) | Superficial Velocity (m/sec) | Volume % Bed Entrained Per Hr. | Entrainment Losses Ca/S Equiv. (Mole/Mole) | Wt. Percent Input Ca Lost by Entrainment | Remarks |
|---|---|---|---|---|---|---|
| 19.7 | 4.0 | 2.1 | 3.3 | 0.60 | 15 | Based on a flyash sample representative of the entire run |
| 20.1 | 3.3 | 2.06 | 5.8 | 1.1 | 32 | |
| 21 | 3.3 | 2.1 | 3.6 | 0.80 | 25 | |
| 30.2 | 3.3 | 2.5 | 1.3 | 0.32 | 10 | Based on flyash samples taken at one-hour intervals |
| | | | 1.2 | 0.30 | 9 | |
| | | | 0.9 | 0.24 | 7 | |
| 32.1 | 3.2 | 2.2 | 1.6 | 0.46 | 14 | |
| | | | 1.3 | 0.38 | 12 | |
| 38.3 | 2.5 | 1.56 | 0.5 | 0.20 | 8 | |
| | | | 0.4 | 0.15 | 6 | |
| | | | 0.4 | 0.17 | 7 | |
| 38.4 | 2.5 | 1.58 | 0.4 | 0.15 | 6 | |
| | | | 0.4 | 0.14 | 6 | |
| | | | 0.3 | 0.12 | 5 | |
| | | Avg. | 0.8 ± 0.5 | 0.24 ± 0.11 | 8 ± 3 | Runs 19.7, 20.1 and 21 are omitted from average |

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Pressure - 640 kPa except Run 32.1 which was 600 kPa
Temperature - 950°C

TABLE F. ENTRAINMENT RATES FOR PFIZER NO. 1337 DOLOMITE

| Miniplant Run No. | Ca/S (Mole/Mole) | Volume Percent Bed Entrained Per Hr. | Entrainment Losses Ca/S Equiv. (Mole/Mole) | Wt. Percent Input Ca Lost by Entrainment | Remarks |
|---|---|---|---|---|---|
| 27.8 | 1.8 | 5.5 | 0.75 | 40 | |
| 27.10 | 1.05 | 1.85 | 0.24 | 23 | |
| 27.13 | .6 | 2.4 | 0.34 | 57 | |
| 27.14 | .7 | 1.5 | 0.21 | 30 | Based on flyash samples taken at one to three hour intervals |
| 27.16 | 1.1 | 3.1 | 0.46 | 42 | |
| 27.17 | 1.1 | 5.5 | 0.82 | 73 | |
| 32.2 | 1.5 | 5.6 | 1.1 | 73 | |
| | | 3.6 | 0.7 | 47 | |
| 32.3 | 1.5 | 3.6 | 0.65 | 38 | |
| | | 3.9 | 0.71 | 47 | |
| | Avg. | 3.7 | 0.6 | 46 | |

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Pressure - 930 kPa except runs 32.2 and 32.3 which were at 600 kPa

Superficial Velocity - 1.5-2.1 m/sec

Temperature - 825-850°C

## TABLE C. PARTICLE SIZE DISTRIBUTION - MINIPLANT USED LIMESTONE NO. 1359 SORBENT

| Run No. | Material | Particle Size (μm), At Indicated Points in Distribution | | | | |
|---|---|---|---|---|---|---|
| | | 10% | 25% | 50% | 75% | 90% |
| 19.3 | Final Bed (1) | 650 | 1100 | 1490 | 2100 | 2460 |
| 19.3 | Rejected Solids (2) | 1000 | 1340 | 1820 | 2200 | 2400 |
| 19.4 | Final Bed | 790 | 1000 | 1340 | 1825 | 2180 |
| 19.4 | Rejected Solids | 880 | 1080 | 1420 | 1900 | 2270 |
| 19.5 | Rejected Solids | 910 | 1110 | 1490 | 1920 | 2190 |
| 19.6 | Rejected Solids | 940 | 1050 | 1370 | 1750 | 2120 |
| 19.7 | Final Bed | 770 | 950 | 1120 | 1630 | 2010 |
| 19.7 | Rejected Solids | 800 | 980 | 1250 | 1760 | 2080 |
| 19.9 | Final Bed | 290 | 480 | 805 | 1330 | 1860 |
| 19.9 | Rejected Solids | 880 | 1150 | 1630 | 2040 | 2300 |
| 20.1 | Final Bed | 670 | 800 | 1020 | 1330 | 1800 |
| 20.1 | Rejected Solids | 740 | 1000 | 1360 | 1840 | 2200 |
| 20.2 | Final Bed | 710 | 880 | 1270 | 1540 | 1960 |
| 20.2 | Rejected Solids | 780 | 970 | 1310 | 1780 | 2140 |
| 21 | Final Bed | 690 | 820 | 1140 | 1650 | 2040 |
| 21 | Rejected Solids | 750 | 930 | 1280 | 1650 | 2180 |
| 22 | Final Bed | 790 | 1010 | 1320 | 1770 | 2110 |
| 26 | Final Bed | 280 | 440 | 860 | 1240 | 1710 |
| 26 | Rejected Solids | 410 | 730 | 1100 | 1620 | 2040 |
| 29 | Final Bed | 310 | 890 | 1250 | 1730 | 2170 |
| 30.1 | Rejected Solids | 680 | 960 | 1220 | 1620 | 2080 |
| 30.2 | Rejected Solids | 750 | 980 | 1220 | 1530 | 1920 |
| 30.3 | Rejected Solids | 950 | 1160 | 1480 | 1960 | 2260 |
| 30.4 | Final Bed | 720 | 950 | 1180 | 1580 | 2000 |
| 39 | Final Bed | 630 | 850 | 1140 | 1580 | 1980 |

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(1) Sorbent in Combustor at End of Run

(2) Sorbent Removed from Combustor During Run

## TABLE 8. PARTICLE SIZE DISTRIBUTION - MINIPLANT USED DOLOMITE NO. 1337 SORBENT

| Run No. | Material | Particle Size (μm), At Indicated Points in Distribution | | | | |
|---|---|---|---|---|---|---|
| | | 10% | 25% | 50% | 75% | 90% |
| 25 | Final Bed (1) | 820 | 1100 | 1540 | 1960 | 2200 |
| 27.3 | Rejected Solids(2) | 640 | 960 | 1430 | 1920 | 2180 |
| 27.5 | Rejected Solids | 340 | 960 | 1580 | 1920 | 2140 |
| 27.8 | Rejected Solids | 170 | 360 | 1120 | 1760 | 2020 |
| 27.10 | Rejected Solids | 260 | 570 | 1220 | 1830 | 2120 |
| 32.3 | Final Bed | 330 | 720 | 960 | 1280 | 1790 |
| 32.3 | Rejected Solids | 440 | 590 | 800 | 1000 | 1260 |
| 34 | Bed 1st Analysis | 160 | 230 | 450 | 960 | 1400 |
| 34 | Bed 2nd Analysis | 170 | 230 | 460 | 1000 | 1480 |
| 35 | Final Bed | 250 | 430 | 830 | 1380 | 1720 |
| 36.2 | Final Bed | 400 | 760 | 1420 | 1930 | 2260 |
| 37 | Final Bed | 240 | 500 | 980 | 1480 | 1860 |
| 38.6 | Final Bed | 170 | 250 | 480 | 1070 | 1780 |

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(1) Sorbent in Combustor at End of Run

(2) Sorbent Removed from Combustor During Run

TABLE 1. PARTICLE SIZE DISTRIBUTION -
MINIPLANT SECONDARY CYCLONE CAPTURE

| Run No. | Particle Size (µm) at Indicated Points in Size Distribution | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | 10% | 20% | 30% | 40% | 50% | 60% | 70% | 80% | 90% |
| 19.2 | 7 | 13 | 20 | 25 | 30 | 48 | 62 | 97 | 150 |
| 19.3 | 7 | 11 | 16 | 22 | 33 | 50 | 64 | 95 | 160 |
| 19.4 | 4 | 6 | 9 | 13 | 19 | 28 | 45 | 76 | 140 |
| 19.5 | 6 | 9 | 13 | 18 | 25 | 36 | 55 | 90 | 180 |
| 19.6 | 6 | 11 | 15 | 20 | 25 | 35 | 50 | 86 | 145 |
| 19.7 | 8 | 13 | 20 | 32 | 48 | 71 | 100 | 159 | 300 |
| 19.9 | 5 | 7 | 10 | 12 | 14 | 17 | 23 | 33 | 52 |
| 20.1 | 8 | 15 | 44 | 46 | 50 | 52 | 54 | 57 | 60 |
| 20.2 | 7 | 10 | 13 | 17 | 22 | 30 | 46 | 90 | 290 |
| 21 | 8 | 15 | 25 | 30 | 37 | 44 | 54 | 80 | 180 |
| 22 | 6 | 9 | 12 | 17 | 24 | 33 | 50 | 75 | 150 |
| 23 | 5 | 7 | 9 | 11 | 15 | 22 | 35 | 59 | 110 |
| 25 | 7 | 12 | 17 | 25 | 38 | 56 | 80 | 120 | 190 |
| 26 | 5 | 8 | 11 | 14 | 17 | 22 | 32 | 46 | 88 |
| 27.1 | 6 | 8 | 11 | 14 | 18 | 21 | 27 | 37 | 56 |
| 27.2 | 5 | 7 | 9 | 11 | 14 | 19 | 25 | 35 | 56 |
| 27.3 | 6 | 8 | 11 | 14 | 18 | 22 | 27 | 37 | 56 |
| 27.4 | 5 | 7 | 10 | 13 | 17 | 21 | 27 | 37 | 58 |
| 27.5 | 6 | 9 | 12 | 16 | 21 | 28 | 37 | 51 | 62 |
| 27.8 | 6 | 9 | 12 | 16 | 21 | 28 | 37 | 51 | 62 |
| 27.9 | 17 | 30 | 46 | 72 | 110 | 160 | 226 | 290 | 320 |
| 27.10 | 5 | 7 | 10 | 12 | 16 | 22 | 31 | 44 | 70 |
| 27.11 | 5 | 9 | 12 | 16 | 20 | 24 | 31 | 43 | 70 |
| 27.13 | 5 | 7 | 9 | 12 | 15 | 19 | 27 | 40 | 70 |
| 27.14 | 6 | 10 | 15 | 19 | 24 | 28 | 35 | 46 | 80 |
| 27.15 | 6 | 9 | 12 | 16 | 20 | 25 | 30 | 40 | 50 |
| 27.16 | 5 | 7 | 10 | 12 | 16 | 21 | 27 | 48 | 54 |
| 27.17 | 5 | 7 | 10 | 12 | 16 | 21 | 27 | 48 | 54 |

TABLE 7 (Continued). PARTICLE SIZE DISTRIBUTION -
MINIPLANT SECONDARY CYCLONE CAPTURE

| Run No. | Particle Size (μm) At Indicated Points in Size Distribution | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | 10% | 20% | 30% | 40% | 50% | 60% | 70% | 80% | 90% |
| 28.1 | 4 | 7 | 11 | 13 | 17 | 21 | 30 | 42 | 66 |
| 28.2 | 4 | 7 | 10 | 13 | 17 | 22 | 31 | 42 | 65 |
| 28.3 | 5 | 7 | 10 | 13 | 17 | 22 | 31 | 45 | 73 |
| 28.4 | 5 | 8 | 12 | 15 | 20 | 26 | 35 | 50 | 75 |
| 28.5 | 5 | 8 | 11 | 14 | 18 | 24 | 32 | 49 | 73 |
| 29.1 | 5 | 9 | 10 | 16 | 21 | 29 | 40 | 53 | 77 |
| 30.1 | 5 | 8 | 11 | 14 | 18 | 25 | 38 | 52 | 78 |
| 30.2 | 5 | 9 | 11 | 14 | 18 | 21 | 29 | 43 | 74 |
| 30.3 | 6 | 10 | 14 | 19 | 26 | 33 | 42 | 54 | 74 |
| 30.4 | 5 | 7 | 8 | 10 | 12 | 16 | 21 | 34 | 64 |
| 31 | 5 | 8 | 10 | 12 | 14 | 18 | 23 | 36 | 64 |
| 32.1 | 7 | 9 | 11 | 13 | 16 | 20 | 26 | 43 | 78 |
| 32.2 | 7 | 10 | 12 | 14 | 16 | 20 | 28 | 43 | 72 |
| 32.3 | 5 | 7 | 9 | 11 | 13 | 16 | 27 | 35 | 64 |

TABLE 1. PARTICLE SIZE DISTRIBUTION -
MINIPLANT FLUE GAS PARTICULATES

| Run Number | Particle Size (μm), At Indicated Points in Distribution | | | | | | |
|---|---|---|---|---|---|---|---|
| | 5% | 10% | 25% | 50% | 75% | 90% | 95% |
| 31 | 1.43 | 1.87 | 3.55 | 8.6 | 23.5 | ~50 | ~60 |
| 32.2 | 1.48 | 1.95 | 3.1 | 5.8 | 12 | 24 | 37.2 |
| 32.3 | 1.75 | 2.3 | 3.3 | 5.9 | 13 | 32.5 | 44 |
| 33 | 2.25 | 3.3 | 4.75 | 7.55 | 13.5 | 25.5 | 42 |
| 36.2 | 1.7 | 2.5 | 4.1 | 7.5 | 14.3 | 30 | 43 |
| 37 | 1.5 | 2 | 3.25 | 4.5 | 14.5 | 30 | 43 |

TABLE 6. MINIPLANT SOLIDS ANALYSES

| | | Weight Percent | | | | |
|---|---|---|---|---|---|---|
| Run No. | Source | Ca | S | $SO_4$ | $CO_2$ | Total C |
| 29.2 | Final Bed (1) | 24 | 4.68 | 22.6, 22.7, 22.97, 23.0 | 20.89 | N.A. (4) |
| | Second Cyclone (2) | 9.3, 9.61, 10.2 | 1.40 | 5.96 | 1.72 | 22.52, 22.61 |
| | Rejected Solids(3) | 32.1 | 1.92 | 18.24 | 6.37 | N.A. |
| 29.3 | Final Bed | 23.4, 23.9 | 7.27 | 20.8 | 29.8 | 6.25 |
| | Second Cyclone | 4.3, 8.26 | 2.89 | 8.4 | .66 | 27.9 |
| | Rejected Solids | 23.3 | 7.7 | 20.4 | 30.2 | 3.67 |
| 29.4 | Final Bed | 23.4, 32.2 | 4.3, 4.17, 4.7 | 18.65 | 35.30 | 7.73 |
| | Second Cyclone | 4.77 | 3.6, 3.29 | 8.97 | 1.69 | 24.90 |
| | Rejected Solids | 34.8 | 5.3, 5.89 | 22.08 | 32.22 | 6.59 |
| 29.5 | Final Bed | 37.8, 37.3, 34.5 | 3.7, 4.46 | 29.74 | 32.98 | 6.46 |
| | Second Cyclone | 9.86, 7.34, 7.64 | 2.2, 2.38 | 6.08 | 1.42 | 22.38 |
| | Rejected Solids | 33.4 | 3.78 | 18.88 | 33.62 | 6.92 |
| 29.6 | Final Bed | 36.4, 33.7 | 3.7, 1.84 | 15.29 | 40.43 | 8.0 |
| | Second Cyclone | 10.1, 8.02, 9.76 | 2.2, 2.26 | 7.34 | 2.37 | 25.85 |
| | Rejected Solids | 35.0 | 4.8 | 29.58 | 33.87 | 6.88 |
| 29.7 | Final Bed | 43.2, 47.3 | 7.3, 5.72 | 21.79 | 8.68 | .37 |
| | Second Cyclone | 26.8, 17.2 | 2.8, 2.91 | 8.27 | 1.38 | 17.30 |
| | Rejected Solids | 40.1 | 5.97 | 20.45 | 12.18 | 2.87 |

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(1) Spent Sorbent Removed from Combustor After End of Run
(2) Solids Captured in Secondary Cyclone
(3) Spent Sorbent Removed from Combustor During Run
(4) Not analyzed

## TABLE X (Continued). MINIPLANT SOLIDS ANALYSES

| Run No. | Source | Weight Percent |  |  |  |  |
|---|---|---|---|---|---|---|
|  |  | Ca | S | $SO_4$ | $CO_2$ | Total C |
| 19.9 | Final Bed | 37.8, 39.5 | 8.0, 10.5 | 29.4 | 2.37 | 1.17 |
|  | Secondary Cyclone | 13.2, 12.8 | 3.74 | 11.46 | 1.19 | 17.44 |
|  | Rejected Solids | 36 | 9.09 | 25.5 | 35.7 | 4.56 |
| 20.1 | Final Bed | 47.5, 45.7 | 8.11 | 23.68 | .51 | N.A. |
|  | Secondary Cyclone | 11.9, 13.8 | 4.72 | 9.98 | 2.51 | 11.5 |
|  | Rejected Solids | 33.2 | 5.98 | 17.9 | 13.1 | N.A. |
| 20.2 | Final Bed | 40.2, 37.8 | 7.41 | 22.4 | .78 | N.A. |
|  | Secondary Cyclone | 13.8, 15.4 | 7.61 | 12.28 | 1.98 | 7.07 |
|  | Rejected Solids | 33.1, 59.3 | 7.98 | 19 | 13.1 | N.A. |
| 21 | Final Bed | 45.2, 46.7 | 7.48 | 20.95 | 23.1 | N.A. |
|  | Secondary Cyclone | 17.7, 17.4 | 3.77 | 10.91 | 4.3 | 12 |
|  | Rejected Solids | 37 | 6.87 | 11.08 | 16 | N.A. |
| 22 | Final Bed | 38, 31.7, 39.5 | N.A. | 30.7 | 18.8 | N.A. |
|  | Secondary Cyclone | 14.7, 12.5, 13.9 | 4.11 | 11.46 | 2.74 | 18.93, 19.45 |
| 23 | Final Bed | 21.7 | N.A. | 29.57 | 16.78 | N.A. |
|  | Secondary Cyclone | 10.2 | 3.93 | 10.76 | 1.22 | 15.24 |
| 25 | Final Bed | 25.7 | 5.95 | 17.18 | 28.5 | N.A. |
|  | Secondary Cyclone | 17.1 | 2.76 | 7.24 | 14.9 | 16.2 |
| 26 | Final Bed | 26.6, 35.4, 32.5 | 9.3 | 27.34 | 19.02 | N.A. |
|  | Secondary Cyclone | 13.9, 11.9 | 4.82 | 8.4 | 4.2 | 13.06 |
|  | Rejected Solids | 32.9 | 11.6 | 24.6 | 5.65 | 2.03 |
| 27.1 | Final Bed | 19.4 | 13.2 | 40.05 | 1.44 | .76 |
|  | Sec. Cyclone #1 | 7.1, 6.04, 6.71 | 3.2 | 9.17 | 0.79 | 19.78 |
|  | Sec Cyclone #2 | 6.1 | 3.42 | 9.28 | 0.58 | 5.75 |

## TABLE K (Continued). MINIPLANT SOLIDS ANALYSIS

Weight Percent

| Run No. | Source | Ca | S | SO4 | CO2 | Total C |
|---|---|---|---|---|---|---|
| 27.2 | Secondary Cyclone | 6.7, 8.07 | 3.7 | 14.63 | .82 | 16.29 |
| | Rejected Solids | 29.5, 28.9 | 10.5 | 30.26 | 11.83 | N.A. |
| 27.3 | Secondary Cyclone | 4.8, 4.7 | 2.2 | 6.71 | .03 | 16.55 |
| | Rejected Solids | 28.6, 24.2, 31.9 | 8.7 | 29.83 | 21.55 | N.A. |
| 27.4 | Secondary Cyclone | 3.5, 3.58 | 1.98 | 4.95 | .63 | 20.94 |
| | Rejected Solids | 29.3, 24.3 | 10.3 | 30.34 | 13.28 | 2.78 |
| 27.5 | Secondary Cyclone | 12.8, 5.65, 8.65 | 5.33 | 15.48 | 5.48 | 12.06 |
| | Rejected Solids | 19.8, 21.9 | 8.4 | 26.11 | 16.08 | 2.26 |
| 27.8 | Secondary Cyclone | 14.1, 26.6 | 7.0 | 9.94 | 4.13 | 9.23 |
| | Rejected Solids | 22.1, 30.7 | 8.9 | 28.94 | 14.45 | .99 |
| 27.9 | Secondary Cyclone | 16.1, 17.8 | 7.4 | 22.10 | 2.69 | 9.26 |
| | Rejected Solids | 23.8, 31.7 | 8.3 | 25.41 | 16.66 | 4.61 |
| 27.10 | Secondary Cyclone | 6.4, 3.48, 5.72 | 2.9 | 16.47 | 0.98 | 18.00 |
| | Rejected Solids | 23.9, 24.6 | 10.31 | 32.54 | 4.46 | 2.17 |
| 27.11 | Secondary Cyclone | 6.1, 9.08, 8.65 | 3.9 | 13.66 | 0.93 | 9.15 |
| 27.13 | Secondary Cyclone | 7.7, 7.44 | 1.3 | 10.57 | .07 | 14.74 |
| | Rejected Solids | 19.6, 12.8 | 9.44 | 32.54 | 3.70 | 2.08 |
| 27.14 | Secondary Cyclone | 6.2, 4.44, 5.7 | 7.28 | 15.43 | 7.42 | 12.22 |
| | Rejected Solids | 21.6, 22 | 12.2 | 30.13 | 6.56 | 2.42 |
| 27.15 | Rejected Solids | 23.4, 28.2 | 11.0 | 31.76 | 12.62 | N.A. |

TABLE X (Continued). MINIPLANT SOLIDS ANALYSIS

| Run No. | Source | Weight Percent<br>Ca | S | $SO_4$ | $CO_2$ | Total C |
|---|---|---|---|---|---|---|
| 27.16 | Secondary Cyclone | 8.33, 8.87 | 2.38 | 14.90 | N.A. | 12.81 |
| 27.17 | Final Bed | 19.4 | 13.2 | 40.05 | 1.44 | .76 |
| | Secondary Cyclone | 15.7, 15.7, 14.9 | 6.4 | 19.05 | 1.19 | 5.17 |
| | Rejected Solids | 19.5 | 10.0 | 30.10 | 1.95 | 2.13 |
| 28.1 | Final Bed | N.A. | 3.1 | N.A. | N.A. | N.A. |
| | Secondary Cyclone | N.A. | 0.53 | N.A. | N.A. | 9.28 |
| 28.2 | Secondary Cyclone | N.A. | 1.15 | N.A. | N.A. | 13.22 |
| 28.3 | Secondary Cyclone | N.A. | 0.55 | N.A. | N.A. | 7.19 |
| 28.4 | Secondary Cyclone | N.A. | 0.50 | N.A. | N.A. | 6.47 |
| 29 | Final Bed | 32.4 | 5.3 | 15.90 | 34.59 | 7.46 |
| | Secondary Cyclone | 6.74 | 3.9 | 8.47 | 0.57 | 11.89 |
| | Rejected Solids | N.A. | N.A. | 18.13 | N.A. | 4.43 |
| 30.1 | Sec. Cyclone #1 | 5.40 | 2.73 | 6.25 | .78 | 16.98 |
| | Sec. Cyclone #2 | 5.68 | 2.80 | 7.26 | .81 | 26.57 |
| | Sec. Cyclone #3 | 6.05 | 3.0 | 8.64 | .55 | 17.84 |
| | Rejected Solids | 30.0 | 7.89 | 23.9 | 28.55 | 5.30 |
| 30.2 | Sec. Cyclone #1 | 9.70 | 2.90 | 8.78 | .62 | 8.90 |
| | Sec. Cyclone #2 | 10.0 | 3.26 | 9.4 | .94 | 11.44 |
| | Sec. Cyclone #3 | 9.31 | 3.2 | 9.28 | .65 | 7.72 |
| | Rejected Solids | 42.3 | 8.8 | 27.25 | .94 | .34 |

TABLE K (Continued). MINIPLANT SOLIDS ANALYSIS

Weight Percent

| Run No. | Source | Ca | S | $SO_4$ | $CO_2$ | Total C |
|---|---|---|---|---|---|---|
| 30.3 | Sec. Cyclone #1 | 6.20 | 3.02 | 7.08 | .85 | 12.98 |
| | Sec. Cyclone #2 | 5.63 | 2.5 | 8.46 | .28 | 15.86 |
| | Sec. Cyclone #3 | 6.27 | 2.33 | 6.15 | .55 | 16.16 |
| | Rejected Solids | 30.2 | 9.06 | 28.19 | 23.49 | 4.24 |
| 30.4 | Final Bed | 33.8 | 3.43 | 9.7 | 44.75 | 7.22 |
| | Sec. Cyclone #1 | 5.97 | 2.6 | 4.8 | .71 | 17.68 |
| | Sec. Cyclone #2 | 5.27 | 2.27 | 6.67 | .75 | 18.40 |
| | Sec. Cyclone #3 | 5.36 | 2.4 | 6.8 | .45 | 21.17 |
| | Rejected Solids | 31.9 | 7.54 | 23.05 | 31.86 | N.A. |
| 31 | Final Bed | 38.4 | 6.30 | 20.24 | 27.90 | 5.63 |
| | Sec. Cyclone #1 | 7.41 | 2.7 | 7.62 | 1.34 | 17.69 |
| | Sec. Cyclone #2 | 6.22 | 2.2 | 6.88 | 1.27 | 19.19 |
| | Rejected Solids | 33.2 | 6.83 | 21.81 | 12.11 | 2.95 |
| 32.1 | Sec. Cyclone #1 | 12.5 | 2.3 | 7.14 | 1.46 | 7.13 |
| | Sec. Cyclone #2 | 12.0 | 1.72 | 5.96 | .69 | 5.56 |
| | Sec. Cyclone #3 | 11.9 | 1.8 | 7.00 | .63 | 5.60 |
| | Rejected Solids | 40.1 | 7.6 | 23.1 | 2.80 | .96 |
| 32.2 | Sec. Cyclone #1 | 18.5 | 6.2 | 17.4 | 3.34 | 3.50 |
| | Sec. Cyclone #2 | 16.7 | 5.93 | 17.2 | 1.72 | 3.42 |
| | Sec. Cyclone #3 | 16 | 4.76 | 14.3 | 1.76 | 3.58 |
| 32.3 | Final Bed | 28.8 | 8.5 | 29.37 | 14.78 | 3.04 |
| | Sec. Cyclone #1 | 10.3 | 5.48 | 10.3 | 2.25 | 13.54 |
| | Sec. Cyclone #2 | 11.6 | 4.08 | 13.5 | 1.73 | 10.50 |
| | Sec. Cyclone #3 | 7.63 | 2.98 | 8.5 | 1.80 | 13.65 |
| 33 | Secondary Cyclone | 22.5 | 5.3 | 15.14 | 5.20 | 1.53 |

TABLE K (Continued). MINIPLANT SOLIDS ANALYSIS

| | | Weight Percent | | | | |
|---|---|---|---|---|---|---|
| Run No. | Source | Ca | S | $SO_4$ | $CO_3$ | Total C |
| 34 | Final Bed | 22.2 | 10.2 | 33.52 | .52, .70 | N.A. |
| | Sec. Cyclone #1 | 13.6 | 7.0 | 20.21 | .46 | 2.20 |
| | Sec. Cyclone #2 | 12.7 | 5.7 | 18.03 | .54 | 4.06 |
| | Sec. Cyclone #3 | 13.1 | 5.9 | 17.70 | .57 | 3.11 |
| 35 | Final Bed | 24.6 | 13.5 | 40.81 | 7.12 | N.A. |
| | Sec. Cyclone #1 | 7.69 | 3.7 | 12.60 | .38 | 6.09 |
| | Sec. Cyclone #2 | 7.54 | 3.8 | 10.64 | .96 | 7.25 |
| | Sec. Cyclone #3 | 8.15 | 3.6 | 11.10 | .18 | 7.36 |
| 36.1 | Sec. Cyclone #1 | 7.28 | 3.0 | 10.06 | .89 | 6.90 |
| | Sec. Cyclone #2 | 7.77 | 3.9 | 10.50 | .70 | 6.85 |
| | Sec. Cyclone #3 | 7.29 | 3.7 | 9.70 | .74 | 6.27 |
| 36.2 | Final Bed | 26.3 | 12.0 | 36.54 | 11.55 | N.A. |
| | Sec. Cyclone #1 | 17.0 | 8.0 | 22.20 | 1.67 | 2.10 |
| | Sec. Cyclone #3 | 15.0 | 6.7 | 20.17 | 1.80 | 3.60 |
| 37 | Final Bed | 21.5 | 14.3 | 46.34 | 2.30 | N.A. |
| | Sec. Cyclone #1 | 12.9 | 8.02 | 24.12 | .81 | 7.35 |
| | Sec. Cyclone #2 | 11.0 | 7.07 | 20.70 | 1.01 | 2.69 |
| | Sec. Cyclone #3 | 11.7 | 7.40 | 24.00 | .71 | 3.78 |
| 38.1 | Sec. Cyclone #1 | 6.56 | 3.66 | 10.75 | 1.72 | 5.10 |
| | Sec. Cyclone #2 | 6.69 | 3.4 | 9.60 | 1.26 | 4.52 |
| | Sec. Cyclone #3 | 5.51 | 3.0 | 8.97 | 1.15 | 5.36 |
| 38.2 | Sec. Cyclone #1 | 5.82 | 3.22 | 9.53 | .93 | 5.19 |
| | Sec. Cyclone #2 | 7.01 | 3.45 | 12.29 | .94 | 5.40 |
| | Sec. Cyclone #3 | 7.49 | 3.76 | 11.86 | 1.02 | 4.89 |

TABLE K (Continued). MINIPLANT SOLIDS ANALYSIS

| Run No. | Source | Weight Percent |  |  |  |  |
|---|---|---|---|---|---|---|
|  |  | Ca | S | $SO_4$ | $CO_2$ | Total C |
| 38.3 | Sec. Cyclone #1 | 9.01 | 4.63 | 14.48 | 2.98 | 17.96 |
|  | Sec. Cyclone #2 | 9.34 | 4.94 | 15.39 | 2.88 | 12.61 |
|  | Sec. Cyclone #3 | 8.68 | 5.9 | 14.74 | 2.66 | 12.55 |
| 38.4 | Sec. Cyclone #1 | 10.1 | 5.46 | 16.34 | 6.10 | 11.22 |
|  | Sec. Cyclone #2 | 11.1 | 5.60 | 16.70 | 6.85 | 12.11 |
|  | Sec. Cyclone #3 | 12.4 | 5.74 | 16.52 | 7.09 | 12.83 |
| 38.5 | Secondary Cyclone | 12.6 | 5.70 | 15.69 | 12.82 | 14.76 |
| 38.6 | Final Bed | 22.7 | 10.7 | 35.05 | 14.87 | N.A. |
|  | Secondary Cyclone | 12.5 | 5.64 | 17.23 | 4.40 | 12.49 |
| 39.1 | Sec. Cyclone #1 | 7.84 | 3.19 | 8.22, 8.62 | 2.12, 2.48 | 12.15 |
|  | Sec. Cyclone #2 | 7.85 | 2.95 | 8.09, 7.60 | 2.28, 2.49 | 15.58 |
|  | Sec. Cyclone #3 | 7.94 | 2.97 | 8.47, 8.13 | 2.56, 2.71 | 15.37 |
|  | Rejected Solids | 32.9 | 2.10 | 5.82, 2.86 | 45.52, 51.91 | 10.23, 9.44 |
| 39.2 | Sec. Cyclone #1 | 6.25 | 2.65 | 7.81, 7.19 | 1.07, 1.20 | 5.27 |
|  | Sec. Cyclone #2 | 6.11 | 2.37 | 7.40, 7.69 | 1.03, 1.24 | 6.36 |
|  | Sec. Cyclone #3 | 6.19 | 2.47 | 7.26, 7.14 | 1.07, 1.14 | 6.40 |
|  | Rejected Solids | 31.9 | 2.81 | 8.27, 4.94 | 31.15 | 9.54, 9.52 |
| 39.3 | Sec. Cyclone #1 | 8.71 | 2.83 | 8.42, 8.37 | .72, .66 | 4.26 |
|  | Sec. Cyclone #2 | 8.39 | 2.74 | 8.17, 8.12 | .56, .64 | 3.77 |
|  | Sec. Cyclone #3 | 8.17 | 2.50 | 6.97, 7.58 | .53, .38 | 5.10 |
|  | Rejected Solids | 40.7 | 6.67 | 19.52, 17.30 | 2.40 | .97 |

TABLE K (Continued). MINIPLANT SOLIDS ANALYSIS

| Run No. | Source | Ca | S | SO$_4$ | CO$_2$ | Total C |
|---|---|---|---|---|---|---|
| | | Weight Percent | | | | |
| 38.4 | Final Bed | 47.0 | 7.05 | 22.63 | 0.45 | <0.3 |
| | Sec. Cyclone #1 | 8.47 | 2.46 | 7.75, 7.59 | .30, .31 | 4.53 |
| | Sec. Cyclone #2 | 7.69 | 2.45 | 7.53, 7.36 | .29, .32 | 4.80 |
| | Sec. Cyclone #3 | 7.74 | 2.60 | 8.54, 7.36 | .49, .38 | 4.42 |
| | Rejected Solids | 4.16 | 6.87 | 21.72, 22.40 | 2.44 | 0.67 |

TABLE L. MINIPLANT SOLIDS COMPOSITION

| Run Number | Source | Composition (Wt. %) | | | Sorbent Portion Composition (Mole %) | | |
|---|---|---|---|---|---|---|---|
| | | C | Ash | Sorbent | CaO | $CaCO_3$ | $CaSO_4$ |
| 19.2 | Final Bed (1) | -4 | 37 | 67 | 4 | 58 | 38 |
| | Reject. Solids(2) | -1 | 36 | 65 | 63 | 13 | 24 |
| | Sec. Cyclone(3) | 22 | 58 | 20 | 62 | 12 | 26 |
| 19.3 | Final Bed | 0.3 | 18 | 82 | 7 | 65 | 28 |
| | Reject. Solids | -0.4 | 29 | 72 | -23 | 87 | 37 |
| | Sec. Cyclone | 28 | 56 | 16 | 37 | 7 | 56 |
| 19.4 | Final Bed | 1 | 19 | 80 | -14 | 86 | 28 |
| | Reject. Solids | 0.2 | 9 | 91 | 12 | 62 | 27 |
| | Sec. Cyclone | 25 | 60 | 15 | -2 | 24 | 78 |
| 19.5 | Final Bed | 0.1 | 14 | 86 | 11 | 64 | 25 |
| | Reject. Solids | 0.2 | 13 | 87 | 9 | 67 | 24 |
| | Sec. Cyclone | 22 | 60 | 18 | 58 | 11 | 31 |
| 19.6 | Final Bed | -0.1 | 11 | 89 | -0.4 | 81 | 19 |
| | Reject. Solids | 0.1 | 10 | 90 | 12 | 65 | 23 |
| | Sec. Cyclone | 25 | 54 | 21 | 50 | 17 | 33 |
| 19.7 | Final Bed | .2 | 20 | 80 | 78 | 1 | 21 |
| | Reject. Solids | .4 | 18 | 82 | 59 | 20 | 21 |
| | Sec. Cyclone | 17 | 50 | 33 | 76 | 5 | 19 |
| 19.9 | Final Bed | 1 | 17 | 83 | 66 | 4 | 30 |
| | Reject. Solids | -3 | 5 | 98 | 4 | 66 | 30 |
| | Sec. Cyclone | 17 | 54 | 29 | 57 | 6 | 17 |
| 20.1 | Final Bed | .2 | 20 | 63 | 77 | 1 | 22 |
| | Reject. Solids | 3 | 26 | 71 | 51 | 26 | 23 |
| | Sec. Cyclone | 11 | 56 | 33 | 65 | 10 | 25 |
| 20.2 | Final Bed | 1 | 32 | 67 | 71 | 2 | 27 |
| | Reject. Solids | -2 | 13 | 89 | 65 | 19 | 16 |
| | Sec. Cyclone | 7 | 61 | 32 | 56 | 9 | 35 |
| 21 | Reject. Solids | -2 | 30 | 73 | 59 | 29 | 13 |
| | | 11 | 52 | 37 | 58 | 16 | 26 |

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(1) Spent Sorbent Removed from Combustor After Run
(2) Spent Sorbent Removed from Combustor During Run
(3) Solids Captured in Secondary Cyclone

TABLE L (Continued). MINIPLANT SOLIDS COMPOSITION

| Run Number | Source | Composition (Wt. %) |  |  | Sorbent Portion Composition (Mole %) |  |  |
|---|---|---|---|---|---|---|---|
|  |  | C | Ash | Sorbent | CaO | $CaCO_3$ | $CaSO_4$ |
| 22 | Final Bed | 1 | 13 | 87 | 25 | 37 | 38 |
|  | Sec. Cyclone | 18 | 51 | 31 | 52 | 13 | 35 |
| 23 | Final Bed | 1 | 20 | 79 | -8 | 52 | 57 |
|  | Sec. Cyclone | 15 | 61 | 24 | 48 | 8 | 44 |
| 25 | Sec. Cyclone | 13 | 34 | 53 | 24 | 58 | 18 |
| 26 | Final Bed | 1 | 19 | 80 | 23 | 40 | 36 |
|  | Reject. Solids | 1 | 28 | 71 | 57 | 12 | 31 |
|  | Sec. Cyclone | 12 | 60 | 28 | 51 | 22 | 27 |
| 27.1 | Sec. Cyclone | 15 | 68 | 17 | 33 | 8 | 60 |
| 27.2 | Reject. Solids | 0.3 | 17 | 82 | 30 | 27 | 43 |
|  | Sec. Cyclone | 16 | 59 | 25 | 21 | 7 | 73 |
| 27.3 | Reject. Solids | -0.2 | 13 | 89 | 5 | 51 | 44 |
|  | Sec. Cyclone | 17 | 71 | 12 | 41 | 0.4 | 59 |
| 27.4 | Reject. Solids | 0.1 | 25 | 75 | 5 | 39 | 56 |
|  | Sec. Cyclone | 21 | 70 | 10 | 30 | 12 | 58 |
| 27.5 | Reject. Solids | -1 | 20 | 81 | 3 | 48 | 49 |
|  | Sec. Cyclone | 11 | 60 | 29 | -15 | 42 | 74 |
| 27.8 | Reject. Solids | -2 | 14 | 88 | 0.1 | 44 | 56 |
|  | Sec. Cyclone | 8 | 52 | 40 | 66 | 14 | 20 |
| 27.9 | Reject. Solids | 1 | 11 | 88 | 5 | 49 | 47 |
|  | Sec. Cyclone | 9 | 47 | 44 | 35 | 11 | 54 |
| 27.10 | Reject Solids | 1 | 16 | 83 | 27 | 13 | 60 |
|  | Sec. Cyclone | 18 | 59 | 23 | -22 | 11 | 111 |
| 27.11 | Sec. Cyclone | 9 | 68 | 23 | 21 | 8 | 72 |
| 27.13 | Reject. Solids | 1 | 27 | 71 | 1 | 15 | 84 |
|  | Sec. Cyclone | 15 | 66 | 20 | 41 | 1 | 58 |
| 27.14 | Reject. Solids | 1 | 15 | 84 | -1 | 22 | 79 |
|  | Sec. Cyclone | 12 | 67 | 21 | -23 | 5 | 118 |
| 27.15 | Reject. Solids | 1 | 16 | 83 | 1 | 39 | 61 |
| 27.17 | Reject. Solids | 2 | 26 | 72 | 29 | 7 | 64 |
|  | Sec. Cyclone | 5 | 57 | 38 | 43 | 5 | 52 |

## TABLE 1 (Continued). MINIPLANT SOLIDS COMPOSITION

| Run Number | Source | Composition (Wt. %) C | Ash | Sorbent | Sorbent Portion Composition (Mole %) CaO | $CaCO_3$ | $CaSO_4$ |
|---|---|---|---|---|---|---|---|
| 29 | Final Bed | 1 | 16 | 84 | 8 | 71 | 21 |
| | Sec. Cyclone | 12 | 71 | 17 | 38 | 10 | 52 |
| 30.1 | Reject. Solids | -0.4 | 18 | 83 | 3 | 64 | 33 |
| | Sec. Cyclone | 17 | 69 | 15 | 38 | 8 | 54 |
| 30.2 | Reject. Solids | 0.2 | 17 | 83 | 72 | 2 | 27 |
| | Sec. Cyclone | 9 | 69 | 22 | 56 | 5 | 39 |
| 30.3 | Reject. Solids | -0.6 | 18 | 83 | 9 | 52 | 39 |
| | Sec. Cyclone | 15 | 71 | 15 | 43 | 7 | 50 |
| 30.4 | Final Bed | -2 | 14 | 88 | -0.4 | 88 | 12 |
| | Reject. Solids | 2 | 11 | 87 | 3 | 67 | 30 |
| | Sec. Cyclone | 19 | 68 | 13 | 47 | 8 | 49 |
| 31 | Final Bed | 0.1 | 9 | 91 | 29 | 49 | 22 |
| | Reject. Solids | 1 | 26 | 74 | 48 | 24 | 27 |
| | Sec. Cyclone | 18 | 65 | 17 | 43 | 13 | 44 |
| | Part. (1) | 7 | 79 | 14 | 11 | 14 | 76 |
| 32.1 | Reject. Solids | 0.4 | 23 | 77 | 71 | 5 | 24 |
| | Sec. Cyclone | 6 | 71 | 23 | 72 | 5 | 23 |
| 32.2 | Sec. Cyclone | 3 | 45 | 52 | 50 | 10 | 40 |
| | Part. | 5 | 75 | 20 | 63 | 3 | 33 |
| 32.3 | Final Bed | 0.1 | 10 | 90 | 23 | 34 | 43 |
| | Sec. Cyclone | 12 | 57 | 31 | 43 | 11 | 45 |
| | Part. | 2 | 72 | 26 | 65 | 4 | 32 |
| 33 | Sec. Cyclone | 1 | 28 | 72 | 57 | 15 | 28 |
| | Part. | 3 | 67 | 30 | 65 | 10 | 25 |
| 34 | Sec. Cyclone | 3 | 50 | 47 | 37 | 2 | 61 |
| 35 | Sec. Cyclone | 7 | 65 | 28 | 34 | 4 | 62 |
| 36.1 | Sec. Cyclone | 7 | 68 | 26 | 37 | 7 | 57 |

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(1) Flue Gas Particulate

### TABLE L (Continued). MINIPLANT SOLIDS COMPOSITION

| Run Number | Source | Composition (Wt. %) | | | Sorbent Portion Composition (Mole %) | | |
|---|---|---|---|---|---|---|---|
| | | C | Ash | Sorbent | CaO | $CaCO_3$ | $CaSO_4$ |
| 36.2 | Final Bed | 2 | 4 | 98 | 13 | 29 | 58 |
| | Sec. Cyclone | 2 | 44 | 53 | 38 | 7 | 55 |
| | Part. | 4 | 73 | 23 | 30 | 6 | 64 |
| 37 | Final Bed | 1 | 13 | 87 | 3 | 7 | 90 |
| | Sec. Cyclone | 3 | 48 | 49 | 14 | 5 | 81 |
| | Part. | 4 | 79 | 17 | 35 | 2 | 63 |
| 38.1 | Sec. Cyclone | 5 | 68 | 27 | 20 | 9 | 71 |
| 38.3 | Sec. Cyclone | 14 | 50 | 36 | 10 | 21 | 69 |
| 38.4 | Sec. Cyclone | 11 | 43 | 46 | -0.9 | 39 | 61 |
| 38.5 | Sec. Cyclone | 11 | 36 | 54 | -18 | 67 | 51 |
| 38.6 | Final Bed | -0.2 | 7 | 93 | -8 | 44 | 64 |
| | Sec. Cyclone | 12 | 41 | 48 | 19 | 24 | 58 |
| 39.1 | Reject Solids | 0.1 | 12 | 88 | 2 | 93 | 5 |
| | Sec. Cyclone | 14 | 66 | 20 | 36 | 21 | 43 |
| 39.2 | Reject. Solids | 3 | 18 | 79 | 35 | 57 | 8 |
| | Sec. Cyclone | 6 | 79 | 16 | 38 | 12 | 50 |
| | Part. | 4 | 80 | 16 | -40 | 3 | 137 |
| 39.3 | Reject. Solids | 0.5 | 20 | 79 | 79 | 4 | 17 |
| | Sec. Cyclone | 4 | 77 | 19 | 56 | 5 | 39 |
| 39.4 | Final Bed | 0.9 | 14 | 85 | 79 | 0.6 | 20 |
| | Reject. Solids | 0.2 | 16 | 84 | 77 | 4 | 20 |
| | Sec. Cyclone | 5 | 78 | 18 | 57 | 3 | 40 |

TABLE 9. SUMMARY OF BATCH COMBUSTOR OPERATING CONDITIONS

| Run No. | Sorbent | Pressure, kPa | Superficial Velocity m/s | Settled Bed Depth, m | Bed Temperature °C Avg. | Bed Temperature °C Range | Excess Air, % | Coal,[1] kg/hr | Run Length, hrs |
|---|---|---|---|---|---|---|---|---|---|
| 59 | 1R | 805 | 1.53 | 0.77 | 870 | 840–895 | 21 | 10.44 | 3.00 |
| 60 | Bed from 59 | 805 | 1.44 | — | 865 | 820–895 | 37 | 9.08 | 3.00 |
| 61 | Bed from 60 | 805 | 1.08 | — | 884 | 861–905 | 62 | 5.38 | 2.75 |
| 62 | 1R | 395 | 1.43 | 0.46 | [illegible] | 840–896 | 61 | 3.77 | 4.[illegible] |
| 63 | 1R | 805 | 1.17 | 0.77 | 914 | 870–952 | 22 | 9.42 | 4.50 |
| 64 | Bed from 63 | 805 | 1.15 | — | 925 | 895–959 | 16 | 6.17 | 2.00 |
| 65 | 1R | 805 | 1.19 | 0.77 | 887 | 804–906 | 17 | 8.39 | 4.00 |
| 66 | 1R | 316 | 1.85 | 0.48 | 903 | 876–915 | 33 | 5.90 | 4.00 |
| 71 | 2L | 815 | 1.11 | 0.77 | 870 | 845–890 | 22 | 7.95 | 2.00 |
| 72 | Bed from 71 | 821 | 0.86 | — | 855 | 840–860 | 41 | 3.44 | 2.00 |
| 73 | Bed from 72 | 811 | 0.8[illegible] | — | 875 | 860–890 | 53 | 4.39 | 1.50 |
| 74 | 2R | 821 | 1.20 | 0.77 | 810 | 790–821 | 80 | 5.95 | 1.75 |
| 75 | 2R | 811 | 1.29 | 0.77 | 820 | 770–840 | 48 | 7.85 | 2.00 |
| 76 | Bed from 75 | 815 | 1.09 | 0.40 | 820 | 775–843 | 73 | 5.72 | 2.00 |
| 77 | Bed from 76 | 811 | 1.05 | 0.36 | 830 | 825–840 | 109 | 4.54 | 2.00 |
| 78 | Bed from 77 | 811 | 0.96 | 0.31 | 940 | 915–960 | 86 | 4.22 | 1.50 |
| 79 | 2R | 811 | 1.08 | 0.77 | 930 | 900–957 | 37 | 6.34 | 3.75 |
| 80 | Bed from 79 | 811 | 0.86 | — | 935 | 930–950 | 53 | 4.77 | 2.00 |
| 81 | 2R | 811 | 1.36 | 0.61 | 960 | 950–975 | 61 | 6.81 | 2.00 |
| 82 | 2R | 426 | 1.64 | 0.61 | 925 | 900–960 | 50 | 4.77 | 2.00 |
| 83 | 1C | 821 | 1.04 | 0.61 | 875 | 850–914 | 1 | 9.08 | 2.00 |
| 84 | 1C | 821 | 1.04 | — | 880 | 850–920 | 0 | 9.08 | 1.75 |
| 85 | 1C | 811 | 1.00 | — | 850 | 800–890 | 6 | 8.49 | 2.00 |
| 95 | 1R | 815 | 1.16 | 0.77 | 850 | 822–873 | 57 | 7.491 | 2.00 |
| 96 | Bed from 95 | 822 | 1.13 | — | 830 | 794–862 | 55 | 7.581 | 1.50 |
| 97 | Bed from 96 | 821 | 1.18 | — | 850 | 812–890 | 60 | 7.491 | 2.00 |
| 99 | 1R | 820 | 1.16 | 0.61 | 902 | 873–960 | 61 | 7.131 | 1.50 |

TABLE 9 (Continued). SUMMARY OF BATCH COMBUSTOR OPERATING CONDITIONS

| Run No. | Sorbent | Pressure, kPa | Superficial Velocity m/s | Settled Bed Depth, m | Bed Temperature °C Avg. | Bed Temperature °C Range | Excess Air, % | Coal, kg/hr | Run Length, hrs |
|---|---|---|---|---|---|---|---|---|---|
| 100 | Bed from 99 | 821 | 1.17 | — | 852 | 830–896 | 73 | 6.822 | 1.50 |
| 101 | Bed from 100 | 821 | 1.16 | — | 883 | 875–902 | 104 | 5.602 | 2.00 |
| 102 | 18 | 821 | 1.15 | 0.61 | 838 | 800–840 | 119 | 6.976 | 5.50 |
| 103 | Bed from 102 | 821 | 1.12 | — | 825 | 770–842 | 120 | 6.805 | 2.00 |
| 104 | 3 | 821 | 1.13 | — | 833 | 800–875 | 179 | 6.546 | 0.50 |
| 105 | Bed from 103 | 821 | 1.15 | — | 850 | 810–868 | 114 | 7.046 | 2.50 |
| 106 | Bed from 105 | 821 | 1.14 | — | 850 | 825–855 | 157 | 5.836 | 3.00 |
| 107 | Bed from 106 | 821 | 1.15 | — | 843 | 825–855 | 153 | 6.046 | 5.00 |

TABLE 9 (Continued) SUMMARY OF BATCH COMBUSTOR OPERATING CONDITIONS

NOTES:

Coal: Arkwright Mine, W. Va., 2.6% S, -16 mesh used unless otherwise noted.
S = Arkwright coal screened to remove fines less than 70 mesh.
W = Wyoming coal, 0.7% S.
I = Illinois coal, 4.2% S.

Stone: (1) = Grove limestone, 8 x 25 mesh (BCR No. 1359).
(2) = Tymochtee dolomite, 8 x 25 mesh.
(3) = Alundum
C = Calcined
U = Uncalcined

TABLE 8. SUMMARY OF BATCH COMBUSTOR EMISSIONS DATA

| Run No. | NO (Average) ppm | NO (Average) lb NO2/10^6 Btu | SO2 (Average) ppm | SO2 (Average) lb SO2/10^6 Btu | % SO2 Reduction | SO2 (Final) ppm | SO2 (Final) lb SO2/10^6 Btu | % SO2 Reduction | % Ca Sulfation | Ca/S | Combustion Efficiency (%) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 59 | 190 | 0.30 | 550 | 1.22 | 68.5 | 778 | 1.72 | 55.5 | 33 | 1.7 | 93.3 |
| 60 | 199 | 0.33 | 646 | 1.50 | 62.0 | 722 | 1.68 | 57.5 | 36 | 1.6 | 95.5 |
| 61 | 191 | 0.38 | 402 | 1.20 | 69.0 | 490 | 1.36 | 61.7 | 32 | 1.9 | 98.2 |
| 62 | 220 | 0.43 | 431 | 1.17 | 69.5 | 761 | 2.06 | 46.1 | 11 | 4.2 | 97.1 |
| 63 | 175 | 0.22 | 350 | 0.61 | 92.6 | 940 | 1.70 | 53.2 | 38 | 1.9 | 96.7 |
| 64 | 151 | 0.21 | 596 | 1.12 | 70.5 | 792 | 1.56 | 58.6 | 39 | 1.5 | 96.1 |
| 65 | 231 | 0.34 | 266 | 0.54 | 85.6 | 696 | 1.34 | 67.4 | 28 | 2.4 | 96.9 |
| 66 | 208 | 0.36 | 647 | 1.45 | 61.6 | 911 | 2.05 | 45.6 | 18 | 2.6 | 91.1 |
| 71 | n.a. | — | 123 | 0.25 | 93.3 | 151 | 0.31 | 91.7 | 36 | 3.9 | 97.5 |
| 72 | n.a. | — | 361 | 0.91 | 76.0 | 647 | 1.54 | 59.3 | 21 | 2.8 | |
| 73 | n.a. | — | 535 | 1.45 | 63.5 | 649 | 1.68 | 55.7 | 62 | 0.9 | |
| 74 | 337 | 0.77 | 173 | 0.35 | 85.4 | 278 | 0.85 | 77.5 | 22 | 3.6 | 93.0 |
| 75 | 286 | 0.51 | 267 | 0.67 | 82.4 | 382 | 0.95 | 74.8 | 23 | 3.3 | 93.5 |
| 76 | 221 | 0.47 | 384 | 1.13 | 70.5 | 576 | 1.69 | 55.7 | 35 | 1.6 | |
| 77 | 193 | 0.48 | 560 | 1.96 | 47.7 | 754 | 2.63 | 29.6 | 42 | 0.7 | |
| 78 | 183 | 0.41 | 558 | 1.74 | 54.0 | 654 | 2.06 | 46.1 | 46 | 1.0 | 98.7 |
| 79 | 226 | 0.37 | 755 | 0.90 | 76.4 | 825 | 1.90 | 48.8 | 33 | 1.5 | 98.0 |
| 80 | 161 | 0.36 | 766 | 1.97 | 47.7 | 975 | 2.46 | 34.7 | 43 | 0.8 | |
| 81 | 207 | 0.56 | 230 | 0.62 | 73.5 | 345 | 0.94 | 75.3 | 28 | 2.7 | |
| 82 | 302 | 0.69 | 229 | 0.58 | 86.6 | 367 | 0.68 | 82.1 | 20 | 4.1 | |
| 83 | 140 | 0.17 | 374 | 0.64 | 83.1 | 411 | 0.71 | 81.4 | 21 | 3.9 | 92.9 |
| 84 | 112 | 0.14 | 502 | 0.86 | 77.5 | 670 | 1.15 | 70.0 | 29 | 2.4 | |
| 85 | 122 | 0.16 | 1031 | 1.86 | 51.5 | 1181 | 2.14 | 44.5 | 40 | 1.1 | |
| 95 | 201 | 0.38 | 1214 | 3.3 | 52.3 | 1721 | 4.7 | 32.4 | 6 | 5.5 | 98.8 |
| 96 | 210 | 0.40 | 1576 | 4.2 | 39.2 | 1825 | 4.9 | 30.0 | 8 | 3.8 | |
| 97 | 230 | 0.46 | 1856 | 5.2 | 26.0 | 1952 | 5.4 | 22.2 | 34 | 1.6 | |

TABLE 9 (Continued). SUMMARY OF BATCH COMBUSTOR EMISSIONS DATA

| Run No. | NO (Average) ppm | NO (Average) lb NO$_2$/10$^6$ Btu | SO$_2$ (Average) ppm | SO$_2$ (Average) lb SO$_2$/10$^6$ Btu | % SO$_2$ Reduction | SO$_2$ (Final) ppm | SO$_2$ (Final) lb SO$_2$/10$^6$ Btu | % SO$_2$ Reduction | % Ca Sulfation | Ca/S | Combustion Efficiency (%) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 99 | 239 | 0.48 | 335 | 0.9 | 86.6 | 933 | 2.6 | 62.6 | 26 | 3.8 | |
| 100 | 240 | 0.51 | 983 | 2.9 | 58.6 | 1222 | 3.6 | 48.5 | 18 | 2.7 | 97.7 |
| 101 | 251 | 0.63 | 1086 | 3.0 | 45.6 | 1496 | 5.2 | 25.1 | 28 | 0.9 | |
| 102 | 241 | 0.65 | 0 | 0.0 | 100.0 | 0 | 0.0 | 100.0 | 16 | 10.0 | |
| 103 | 299 | 0.87 | 0 | 0.0 | 100.0 | 0 | 0.0 | 100.0 | 8 | 12.8 | |
| 104 | 174 | 0.49 | 135 | 0.5 | 58.1 | 174 | 0.7 | 46.0 | | | |
| 105 | 261 | 0.69 | 39 | 0.1 | 88.4 | 39 | 0.1 | 88.4 | 9 | 6.9 | |
| 106 | 291 | 0.92 | 19 | 0.1 | 93.3 | 19 | 0.1 | 93.3 | 13 | 7.3 | 98.9 |
| 107 | 272 | 0.86 | 19 | 0.1 | 93.6 | 19 | 0.1 | 93.6 | 12 | 7.7 | |

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n.m. = not measured because of problems with equipment

## TABLE O. BATCH FLUIDIZED BED COMBUSTOR CO EMISSIONS

| Run No. 3675- | CO (ppm) | Temperature (°C) | Excess Air (%) |
|---|---|---|---|
| 59 | 332 | 870 | 21 |
| 60 | NA | 865 | 37 |
| 61 | 201 | 896 | 62 |
| 62 | 287 | 875 | 61 |
| 63 | 164 | 915 | 12 |
| 64 | 118 | 925 | 16 |
| 65 | 170 | 887 | 17 |
| 66 | 360 | 903 | 33 |
| 71 | 104 | 870 | 22 |
| 72 | 167 | 855 | 41 |
| 73 | 167 | 875 | 53 |
| 74 | 174 | 810 | 88 |
| 75 | 136 | 820 | 48 |
| 76 | 125 | 820 | 73 |
| 77 | NA | 830 | 109 |
| 78 | 87 | 940 | 86 |
| 79 | 79 | 930 | 37 |
| 80 | 136 | 935 | 53 |
| 81 | 72 | 960 | 61 |
| 82 | 143 | 925 | 50 |
| 83 | 187 | 875 | 1 |
| 84 | 163 | 880 | 0 |
| 85 | 305 | 850 | 6 |
| 95 | 287 | 850 | 57 |
| 96 | 287 | 830 | 55 |
| 97 | 287 | 850 | 60 |
| 99 | 96 | 902 | 61 |
| 100 | 144 | 882 | 70 |
| 101 | 145 | 88[illegible] | 104 |
| 102 | 97 | 838 | 119 |
| 103 | 121 | 825 | 120 |
| 104 | 242 | 833 | 129 |
| 105 | 121 | 850 | 114 |
| 106 | 121 | 850 | 157 |
| 107 | 97 | 843 | 153 |

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N.A. = Not Analysed

## TABLE F. BATCH COMBUSTOR PARTICLE SIZE DISTRIBUTION - OVERHEAD SAMPLES

| Run No. | Particle Size(um) | Weight Percent Finer Than | |
|---|---|---|---|
| | | Cyclone 1 | Filter |
| 61 | 600 | 99.3 | NA |
| | 300 | 80.7 | NA |
| | 150 | 48.6 | NA |
| | 106 | 37.2 | NA |
| | 75 | 22.9 | NA |
| | 44 | 14.3 | NA |
| 65 | 600 | 94.3 | NA |
| | 300 | 88.6 | NA |
| | 150 | 74.3 | NA |
| | 106 | 68.6 | NA |
| | 75 | 60.0 | NA |
| | 44 | 45.7 | 87.5 |
| 73 | 600 | 99.0 | 96.3 |
| | 300 | 88.4 | 92.6 |
| | 150 | 74.0 | 88.9 |
| | 106 | 66.8 | 87.0 |
| | 75 | 57.7 | 85.1 |
| | 44 | 43.3 | 79.5 |
| 81 | 600 | 99.3 | 100.0 |
| | 300 | 82.9 | 100.0 |
| | 150 | 63.6 | 98.7 |
| | 106 | 56.5 | 98.1 |
| | 75 | 47.2 | 79.2 |
| | 44 | 34.3 | 75.4 |

TABLE Q. BATCH COMBUSTOR BED AND OVERHEAD SOLIDS ANALYSIS

| Run No. | Bed |  |  | Cyclone 1 |  |  |  | Filter |  |  |  |
|---|---|---|---|---|---|---|---|---|---|---|---|
|  | $Ca^{++}$ | $SO_4^{=}$* | $CO_3^{=}$* | Total C^a | Total S | $SO_4^{=}$* | $CO_3^{=}$* | Total C | Total S | $SO_4^{=}$* | $CO_3^{=}$* |
| 59 | 35.30 | 27.86 | 19.98 | 35.46 | 2.96 | NA | 2.25 | 27.25 | 3.95 | NA | 0.52 |
| 60 | 36.60 | 32.60 | 0.97 | 34.02 | 3.75 | NA | 0.66 | 15.86 | 4.60 | NA | 2.26 |
| 61 | 43.50 | 33.16 | 3.13 | 11.32 | 9.31 | NA | 4.07 | NA | NA | NA | NA |
| 62 | 52.30 | 14.05 | 2.39 | 20.00 | 2.42 | NA | 2.21 | NA | NA | NA | NA |
| 63 | 43.00 | 28.86 | 2.93 | 27.69 | 1.40 | NA | 1.07 | 13.80 | 3.50 | NA | 7.21 |
| 64 | 39.70 | 38.37 | 2.17 | 34.81 | 2.06 | NA | 0.50 | 16.10 | 4.73 | NA | 0.12 |
| 65 | 43.80 | 29.76 | 6.01 | 25.11 | 2.39 | NA | 3.58 | 15.17 | 4.41 | NA | 0.86 |
| 66 | 50.00 | 21.05 | 1.34 | 52.51 | 2.62 | NA | 2.52 | 24.44 | 3.43 | NA | 0.83 |
| 71 | 29.20 | 16.32 | 2.35 | NA | NA | NA | NA | NA | NA | NA | NA |
| 72 | 44.60 | 22.52 | 0.95 | 14.52 | 3.60 | NA | 3.32 | 11.13 | 5.40 | NA | 5.70 |
| 73 | 17.30 | 27.17 | 1.27 | NA | NA | NA | NA | NA | NA | NA | NA |
| 74 | 25.50 | 13.12 | NA | 12.83 | 5.00 | NA | NA | 13.42 | 4.10 | NA | NA |
| 75 | 25.00 | 13.59 | 24.65 | NA | NA | NA | NA | NA | NA | NA | NA |
| 76 | 25.08 | 20.56 | 15.53 | 26.60 | 5.30 | NA | 4.02 | 30.31 | 3.10 | NA | 2.96 |
| 77 | 22.90 | 23.66 | 16.68 | NA | NA | NA | NA | NA | NA | NA | NA |
| 78 | 24.90 | 28.16 | 1.76 | 8.91 | NA | NA | 2.83 | 8.91 | NA | NA | NA |
| 79 | 27.40 | 22.35 | 1.97 | 15.97 | 3.00 | NA | 6.53 | 10.70 | 4.50 | NA | 6.87 |
| 80 | 25.40 | 27.25 | 2.33 | NA | NA | NA | NA | NA | NA | NA | NA |
| 81 | 27.60 | 18.10 | 3.50 | NA | NA | NA | NA | NA | NA | NA | NA |
| 82 | 28.90 | 14.03 | 3.71 | NA | NA | NA | NA | NA | NA | NA | NA |
| 83 | 45.20 | 22.99 | 2.21 | NA | NA | NA | NA | NA | NA | NA | NA |
| 84 | 42.90 | 29.47 | 1.96 | 30.74 | 3.10 | NA | NA | 44.92 | 2.90 | NA | NA |
| 85 | 39.60 | 27.41 | 3.66 | NA | NA | NA | NA | NA | NA | NA | NA |
| 95 | 36.90 | 5.19 | 30.75 | NA | NA | NA | NA | NA | NA | NA | NA |
| 96 | 36.10 | 6.74 | 41.11 | 8.11 | 3.46 | 0.64 | 0.96 | 11.23 | 0.20 | 21.05 | 0.07 |
| 97 | 35.10 | 11.60 | 44.60 | NA | NA | NA | NA | NA | NA | NA | NA |
| 99 | 46.40 | 18.33 | 0.32 | NA | NA | NA | NA | NA | NA | NA | NA |
| 100 | -3.20 | 18.51 | 3.54 | 9.55 | NA | 9.60 | 3.87 | 9.26 | 0.14 | 25.60 | 0.53 |
| 101 | 41.30 | 29.13 | 0.76 | NA | 3.37 | NA | NA | NA | NA | NA | NA |
| 102 | 34.90 | 8.17 | 38.64 | 2.85 | 4.27 | 12.34 | 7.33 | 3.13 | 6.38 | 3.82 | 0.23 |
| 103 | 39.20 | 22.09<br>(7.35) | 47.49 |  |  |  |  |  |  |  |  |

TABLE Q (Continued). BATCH COMBUSTOR BED AND OVERHEAD SOLIDS ANALYSIS

| Run | Bed | | | Cyclone 1 | | | | Filter | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| No. | $Ca^{++}$ | $SO_4^=$ | $CO_3^=$ | Total C[a] | Total S | $SO_4^=$ | $CO_3^=$ | Total C | Total S | $SO_4^=$ | $CO_3^=$ |
| 105 | 51.50 | 10.90 | 8.35 | NA | NA | NA | NA | NA | NA | NA | NA |
| 106 | 46.10 | 14.20 | 12.12 | 1.40 | 3.90 | 12.35 | 3.39 | 3.47 | 5.30 | 15.06 | 10.44 |
| 107 | 49.10 | 14.00 | 3.60 | NA | NA | NA | NA | NA | NA | NA | NA |

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All values are weight percent

[a] Includes carbon present as $CO_3^=$

## TABLE 8. SULFUR BALANCES FOR BATCH COMBUSTOR

(All weights in kg)

| Run No. | Sulfur In Coal | Sulfur Out: Flue Gas (1) | Sulfur Out: Overhead Solids | Sulfur Out: Bed | Sulfur Out: Total | % Sulfur Balance |
|---|---|---|---|---|---|---|
| 59<br>60<br>61 | 1.840 | 0.636 | 0.539 | 0.477 | 1.652 | 90 |
| 62 | 0.392 | 0.120 | 0.049 | 0.177 | 0.346 | 88 |
| 63<br>64 | 1.479 | 0.308 | 0.143 | 0.900 | 1.351 | 91 |
| 65 | 0.847 | 0.122 | 0.116 | 0.775 | 1.013 | 120 |
| 66 | 0.595 | 0.230 | 0.120 | 0.290 | 0.640 | 108 |
| 71<br>72<br>73 | 0.863 | 0.173 | 0.232 | 0.411 | 0.816 | 95 |
| 74 | | | | | | |
| 75<br>76<br>77 | 0.922 | 0.286 | 0.386 | 0.377 | 1.049 | 114 |
| 79<br>80 | 0.854 | 0.281 | 0.191 | 0.454 | 0.926 | 108 |
| 83<br>84<br>85 | 1.288 | 0.378 | 0.259 | 1.132 | 1.769 | 137 |
| 95<br>96<br>97 | 1.751 | 1.006 | 0.189 | 0.351 | 1.546 | 88 |
| 99<br>100<br>101 | 1.419 | 1.060 | 0.302 | 0.466 | 1.828 | 129 |
| 105<br>106<br>107 | 0.403 | 0.000 | 0.242 | 0.144 | 0.385 | 96 |

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(1) Based on average $SO_2$ concentration.

Average = 103%
Standard deviation = 17%

TABLE 5. CALCIUM BALANCES FOR BATCH COMBUSTOR

| Run No. | Charge (stone, kg) | Input (kgCa) | Wt. Bed Recovered, kg | Wt. Fr. Ca in Bed | Wt. Ca in Bed kg | Wt. Overhead Solids Recovered, kg | Wt. Fr. Ca in Overhead | Wt. Ca in Overhead | Output (kgCa) | % Calcium Balance |
|---|---|---|---|---|---|---|---|---|---|---|
| 145 | TD, 7.56 | 1.58 | 2.63 | 0.223 | 0.58 | 5.06 | 0.170 | 0.86 | 1.44 | 91 |
| 2C | TD, 7.56 | 1.58 | 2.63 | 0.256 | 0.67 | 4.20 | 0.101 | 0.42 | 1.09 | 69 |
| 3C | G, 7.72 | 3.02 | 4.72 | 0.433 | 2.05 | 4.23 | 0.0899 | 0.38 | 2.43 | 80 |
| 4C | TD, 7.56 | 1.58 | 0.82 | 0.285 | 0.23 | 4.72 | 0.218 | 1.03 | 1.26 | 80 |
| 5C | TD, 7.56 | 1.58 | 4.65 | 0.267 | 1.23 | 1.81 | 0.082 | 0.15 | 1.38 | 87 |
| 17C | G, 7.72 | 3.02 | 5.08 | 0.461 | 2.34 | — | — | 0.24 | 2.58 | 85 |
| 23C | TD, 7.72 | 1.61 | 4.27 | 0.295 | 1.26 | — | — | 0.39 | 1.65 | 102 |
| 95C<br>96C<br>97C | G, 11.35 | 4.44 | 9.08 | 0.351 | 3.19 | — | — |  |  |  |
| 98C<br>100C<br>101C | G, 9.08 | 3.55 | 4.99 | 0.413 | 2.06 | — | — | 0.94 | 3.00 | 85 |
| 102C<br>103C<br>105C<br>106C<br>107C | G, 9.08 | 3.55 | 3.06 | 0.491 | 1.50 | — | — | 2.06 | 3.56 | 100 |

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TD - Tymochtee Dolomite, 20.9 Wt. % Ca

G - Grove Limestone, 39.1 Wt. % Ca